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IS 2796 (2008): Motor Gasolines [PCD 3: Petroleum, Lubricants and their Related Products]

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(चौथा पुनरीक्षण)

Indian Standard

MOTOR GASOLINE — SPECIFICATION

(Fourth Revision)

ICS 43.060.01;75.160.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2008

Price Group 9

Petroleum Lubricants and Their Related Products Sectional Committee, PCD 3

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Gasoline is a complex mixture composed of relatively volatile hydrocarbons that vary widely in their physical and chemical properties. Gasoline is exposed to a wide variety of mechanical, physical and chemical environments. Thus, the properties of gasoline must be balanced to give satisfactory engine performance over an extremely wide range of operating conditions. The performance of gasoline in spark ignition engines depends on a number of factors like engine design, engine maintenance, driving cycle, environment and fuel quality parameters like octane number, volatility, stability, etc.

This standard was first published in 1964 and covered motor gasoline of 83 octane only. Subsequently, it was revised in 1971 by enlarging its scope to cover motor gasoline of 93 octane also keeping in view the increasing demand of 93 octane motor gasoline in the country. Since the test method IS 1448 [P : 37] 'Determination of tetraethyl lead (TEL) by chlorate oxidation method' was withdrawn, the reference to this method was deleted from the standard through Amendment No. 1, October 1979 and the latest test method IS 1448 [P : 82] 'Determination of lead content in gasoline by iodine monochloride method' was introduced. On the recommendations of vehicle manufacturers the octane number requirements of 83 octane motor gasoline were again revised through Amendment No. 2 in November 1984 and the same was upgraded to 87 octane motor gasoline, keeping the other requirements unchanged. Amendment No. 3 in 1992 was issued with a view to provide for use of methanol blended (3 percent *v/v*) motor gasoline, as desired by the Ministry of Petroleum and Natural Gas (MoP&NG) with the purpose of conserving petroleum products and also utilizing the surplus methanol available. The requirement for water tolerance of gasoline-methanol blends was also incorporated. Methanol being known as a highly toxic substance, precautionary note against its safe use was incorporated in the amendment.

The second revision of this standard was published in 1995 in accordance with the requirement of engines available at that time and exhaust emission norms in vogue. The requirement of anti-knock rating (octane number) was replaced by the anti-knock index which is the average of Research Octane Number (RON) and Motor Octane Number (MON), that is, (RON+MON)/2, since anti-knock index is a better representation of the road octane requirements for a vehicle. Vapour-Lock Index (VLI) was introduced in addition to distillation and Reid Vapour pressure to prevent 'vapour-lock' problem in cars under hot weather conditions.

In India with an increasing use of Fluidized Catalytic Cracking (FCC) units in the refineries, the amount of cracked stock in gasoline drastically increased which adversely affected the storage stability and consequently the fuel economy, emission and drivability. However, by appropriate combination of refining process and use of multifunctional additives the deposit formation could be avoided. In the second revision two major requirements were introduced:

a) Requirement of potential gum in place of induction period, and
b) Engine test method for ensuring engine intake system cleanliness (Carburettor, induction manifold and intake valves) as a 'Type Test' to provide trouble-free operation of cars.

Investigations had established that the potential gum corresponding to 4 h of ageing at 100°C and under oxygen pressure of 690 kPa (ASTM D 873) represents gum content after storage for three months at 43°C. The engine evaluation was introduced to establish the carburettor and intake system deposition tendency. This test helped in identifying the potential additive candidates for the type of fuel produced and also to establish and optimize the additive dosage to achieve cleanliness level in fuel and intake system of the engine.

In addition to get octane boost particularly for unleaded gasoline and to incorporate oxygen in fuels, use of organic oxygenates like methyl tertiary butyl ether (MTBE), methanol, ethanol and higher alcohols containing 2.0-3.7 mass percent oxygen (O_2) in gasoline was being practiced in advanced countries. Accordingly, use of organic oxygenates was permitted as blending components or as stabilizing agents to prevent phase separation of the gasoline/alcohol blends. The properties of gasoline-oxygenate blends can differ considerably from those of gasoline alone. Consequently, additional requirements were needed for gasoline-oxygenate blends. These requirements involved evaluation of compatibility with plastics and elastomeric material in fuel systems, corrosion of metals, and especially in the case of gasoline-alcohol blends, water tolerance which was used to indicate the ability of a gasoline-oxygenate blends to dissolve water without the phase separation. The Reid Vapour Pressure Method (wet) was made applicable to gasoline and gasoline-ether blends but not to gasoline-alcohol blends. The dry vapour pressure method as described in Annex A (ASTM D 4953) shall be used for gasoline-alcohol blends. Considering the availability of unleaded motor gasoline in near future, methods of sampling and test for 87 octane unleaded motor gasoline was incorporated.

Amendment No. 1, March, 1997 was issued to reduce the sulphur content for unleaded motor gasoline from 0.20 to 0.15 percent by mass, *Max*. The requirement of sulphur content was aligned with Notification No. GSR 176(E) dated 2.4.1996, issued by the Ministry of Environment and Forests (MoEF).

Amendment No. 2, March, 1999 was issued to restrict the lead content from 0.56 to 0.15 g/l, *Max* for 87 octane leaded grade gasoline.

Amendment No. 3, March, 2000 was issued to introduce the requirement of benzene content as 3 percent by volume, maximum for the metros.

Amendment No. 4, September, 2000 incorporated the requirement of Research Octane Number (RON) in addition to AKI.

The third revision of this standard was published in December 2000 with a view to take into account the requirement of newer engines being introduced and stricter exhaust emission norms being mandated by the Government with penal provisions for contravention, incentives (duty concession) for fuel efficient engine designs and the increasing users awareness to problems of drivability, safety, equipment life and fuel economy. Besides, some of the requirements had been modified as below:

a) Leaded regular grade gasoline deleted and only two grades of unleaded gasoline, namely, regular and premium retained;
b) Requirement of density provided as 710 to 770 kg/m^3;
c) Sulphur content restricted to 0.10 percent, by mass, *Max*;
d) Range of Reid Vapour pressure modified as 35 to 60 kPa;
e) Requirement of benzene content stipulated as 3 percent *(v/v), Max* for metros and 5 percent *(v/v), Max* for the rest of the country; and
f) Provision had been made for use of multifunctional additives (MFA) from internationally accepted test laboratories/authorities in place of evaluation of gasoline engine intake system deposition by twin carburettor Ambassador Gasoline Engine Test.

Subsequently, following five amendments of third revision were issued to incorporate various requirements including the motor gasoline specifications for vehicles meeting Bharat Stage II (BS II) and Bharat Stage III (BS III) emission norms:

Amendment No. 1, January 2002 was issued primarily with the note that the twin carburettor ambassador gasoline engine test may be retained to be employed concurrently up to April 2003 and change in requirement in characteristics of Sulphur, Benzene and Engine intake system cleanliness in Table 1, as well as modifications in the Note 6 of the table pertaining to Multi Functional Additives (MFA) and fuel intake system cleanliness.

Amendment No. 2, February 2003, was issued incorporating the reworking of footnote 6 of Table 1 on the Amendment No. 1 and the Euro III/ Bharat Stage III emission norms compliant specifications for motor gasoline.

Amendment No. 3, July 2003, was issued incorporating the effect of 5 percent (*v/v*), maximum ethanol in Motor Gasoline on RVP, and VLI and their of relaxation. In view of the different brands of motor gasoline launched by marketing companies, the provision was made for different colours under Table 1, Sl No. 1. The test method for analysis of ethanol content was also incorporated.

Amendment No.4, August 2004, was issued on Table 1 footnote 6 for redefining the test methods for assuring adequate fuel system and intake system cleanliness performance of engine.

Amendment No. 5, July 2005 was issued to align the requirement for motor gasoline with auto fuel policy of the Government of India. Addition of methanol to motor gasoline was discontinued. The anhydrous ethanol for use in automotive fuel was also defined in IS 15464. Ministry of Petroleum and Natural Gas (MoP&NG) vide its Gazette Notification extraordinary Part I – Section 1 under SI No. 230 and resolution No. P-45018/28/2000-CC dated 3rd September 2002 mandated sale of only 5 percent ethanol blended motor gasoline with effect from 1st January 2003 in the notified States and Union Territories. However, due to non-availability of adequate ethanol/price differential, the dates of implementations as well as mandate were also amended from time-to-time. Accordingly Table 1 was modified and amended for characteristics, requirements as well as method of tests. Motor Gasoline under the requirement was placed in 2 columns with BS II and BS III with the grades MS 88, MS 93 and MS 91, MS 95 respectively. Provision had been made under Note 7 that MFA which are certified against National Generic Certification Option as per US – EPA – 97 final rule (40 CFR part 80 certification standards for deposits control additives) can also be used by fuel Refiners/ Marketers at treatment level not less than the lowest additive concentration (LAC) limits, as these MFAs meet the criteria for acceptance.

The present version of this standard (fourth revision) has been necessitated with a view to effect some editorial corrections arising in correlating the above mentioned amendments and to incorporate test methods for determination of ethanol content and ISO 4259 for interpretation of test results based on the precision statements of respective test methods. This revision includes all the five amendments issued to the third revision of the standard.

While assessing the quality requirement of motor gasoline, it was felt that it needs overall improvement in order to meet stricter emission norms envisaged in future. Keeping in view that a huge investment and considerable time required to carry out any major process changes in the refineries, the Committee decided to stipulate the quality requirement for motor gasoline for future so as to provide sufficient lead time for refineries. A target specification for the purpose has thus been given in Annex F so that it would serve as guidelines not only for the existing refineries to augment their professing facilities, but also for engine/vehicle manufacturers. Assistance has been derived from the recommendations of Expert Committee on Auto Fuel Policy headed by Dr R.A. Mashelkar, Director General CSIR and Inter-Ministerial Task Force headed by Dr D.K. Biswas, Chairman, CPCB. The requirements envisaged in target specification for motor gasoline would be as per the requirements for vehicles meeting Bharat Stage IV/Euro IV Vehicular Emission Norms. The date and area for implementation of target specification are expected to be decided by the Competent Authority.

It is recognized that there are applications where for technical or other reasons, limits different from those in this standard or additional requirements may be necessary. This standard does not cover such special applications which are matters for agreement between the purchaser and the supplier. This specification, unless otherwise, provided by agreement between the purchaser and the supplier, prescribes the required properties of motor gasoline at the time and place of delivery. Nothing in this specification shall, however, preclude observance of the regulations which may be more restrictive.

An Amendment to incorporate the provision of 10 percent ethanol blending in motor gasoline (E10) in the present version (fourth revision) was initiated after the finalization of this standard, which has since been finalized and approved by the Committee. The details of the same have been incorporated in the main text of the standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 MARCH 2010
TO
IS 2796 : 2008 MOTOR GASOLINE — SPECIFICATION

(Fourth Revision)

[*Foreword, page* (iii), *para* 3 *starting with* 'While'] — Substitute the following for the existing:

'The specification for motor gasoline for the vehicles meeting Bharat Stage IV/Euro IV Vehicular Emissions Norms based on the Auto Fuel Policy, issued by the Ministry of Petroleum and Natural Gas, Government of India has been incorporated in Annex F. Assistance has been derived from the recommendations of the Expert Committee on Auto Fuel Policy headed by Dr R. A. Mashelkar, Director General, CSIR and Inter-Ministerial Task Force headed by Dr D. K. Biswas, Chairman, CPCB. The date and area for implementation of this specification are as decided by the Competent Authority.'

[*Page* 3, *Table* 1, *Sl No.* (xii), *col* 3] — Substitute '35-60' *for* '35-69'.

(*Page* 4, *Table* 1, *Note* 2, *line* 2) — Substitute '4.75 ± 0.25 percent (*v/v*)' *for* '4.75 ± 0.25 (*v/v*)'.

[*Page* 4, *Table* 1, *Note* 7(b), *col* 3] — Substitute 'Chrysler' *for* 'Chrysier'.

[*Page* 6, *Table* 2, *Note* 6(b), *col* 3] — Substitute 'Chrysler' *for* 'Chrysier'.

[*Page* 6, *Table* 2, *Note* 7] — Insert 'and/or water extraction method' *after* '(FTIR)'.

(*Page* 18, *Annex* C, *clause* **C-14.1.1**, *informal table, Under* '*Repeatability*') — Substitute '0.09 ($X^{0.59}$) up to 0.02 ($X^{1.26}$)' *for* '0.09 (*X* 0.59) up to 0.02 (*X* 1.26)'.

(*Page* 18, *Annex* C, *clause* **C-14.1.2**, *informal table, Under* '*Reproducibility*') — Substitute '0.37 ($X^{0.59}$) up to 0.09 ($X^{1.27}$)' *for* '0.37 (*X* 0.59) up to 0.09 (*X* 1.27)'.

(*Page* 20, *Annex* E) — Substitute the following for the existing Annex:

ANNEX E
[*Table* 1, *Sl No.* (xxii)(b), *Note* 8]

METHOD FOR DETERMINATION OF ETHANOL CONTENT IN ETHANOL DOPED MOTOR GASOLINE BY WATER EXTRACTION

E-1 SCOPE

This method covers the determination of ethanol content in ethanol doped Motor Gasoline as field test at supply point, Retail Outlet, etc, by using water. This method provides a test of online ethanol doped gasoline for ethanol content in percentage volume. The method can suitably be used for estimation of ethanol content in ethanol doped Motor Gasoline and quantification of ethanol up to 16 percent as a referee test method. In case of dispute the sample may be subjected to method ASTM D 4815/Annex C.

E-2 OUTLINE OF THE TEST METHOD

30 ml of water is added to 100 ml of ethanol doped Motor Gasoline taken in 150 ml graduated stoppered cylinder. The mixture is shaken vigorously for about 30 seconds and cylinder is set aside for five minutes to observe the separation of aqueous layer. Increase in the volume of aqueous layer over and above 30 ml indicates the presence of extractable ethanol in the ethanol doped motor gasoline sample tested. The exact quantity of ethanol is then worked out referring the observation to the calibration plot. The plot is made on exact quantity of ethanol extracted from 100 ml of various known concentrations of ethanol doped Motor Gasoline using 30 ml of water vis-à-vis the concentration of ethanol used for doping. For better results the calibration chart shall be re-plotted whenever there is change in the source of Motor Gasoline to minimize the effect of solubility coefficient with respect to different hydrocarbon composition of Motor Gasoline.

E-3 SIGNIFICANCE AND USE

The test method is employed to check the percentage of ethanol in ethanol doped Motor Gasoline, as ethanol is having more affinity for mixing with water. The addition of water in ethanol doped Motor Gasoline extracts the

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ethanol present in the ethanol doped Motor Gasoline in to aqueous phase. The increase in the water level indicates the quantity of extractable ethanol in Ethanol Doped Motor Gasoline.

E-4 CHEMICAL REQUIREMENTS

E-4.1 Ethanol/Denatured Ethanol, is the pure chemical, otherwise known as hydroxy-ethane, corresponding to the constitution CH_3CH_2OH and molecular formula, C_2H_5OH. Anhydrous Ethanol for an Automatic fuel (IS 15464) which is made unfit for beverage use by the addition of noxious or toxic materials (denaturants) and same is used as ethanol for the purpose of doping Motor Gasoline.

E-4.2 Motor Gasoline, a volatile mixture of liquid hydrocarbon, generally containing a small amount of additives, suitable for use as a fuel in spark ignition and internal combustion engines conforming to Tables 1 and 2.

E-4.3 Ethanol Doped Motor Gasoline, a fuel consisting primarily of Motor Gasoline doped with denatured anhydrous Ethanol for fuel.

E-4.4 Water, distilled is to be used.

E-5 APPARATUS

E-5.1 150 ml glass stoppered cylinder with 0.5 ml graduation from 0 to 50 ml and 1 ml graduation from 50 to 150 ml having dimensions as per **E-5.2 (***see* Fig. 1).

GRADUATIONS: 0 to 50 ml = 0.5 ml
50 to 150 ml = 1.0 ml

FIG. 1 150 ml GRADUATED STOPPERED CYLINDER

E-5.2 Dimensions of 150 ml Cylinder in Millimetres

Total Length: 340 ± 5
Scale Length: 275 ± 2
Scale Length from (0 ml to 50 ml scale) : 105 ± 2
Scale Length from (50 ml to 150 ml scale) : 170 ± 2
Stopper Joint: B-19

E-5.3 10 ml or 25 ml and 5 ml glass pipette (Class ‘A’ Certificate).

E-6 CALIBRATION ON KNOWN ETHANOL CONTENT IN ETHANOL DOPED MOTOR GASOLINE AND ITS EXTRACTION

E-6.1 Prepare a set of standards of ethanol doped Motor Gasoline samples with the concentration of ethanol in Motor Gasoline ranging from 2.0 to 16.0 ml by percentage volume. Take 100 ml of each sample above in 150 ml graduated stoppered clean and dry glass cylinder along with one blank Motor Gasoline (without ethanol).

Add precisely 30 ml of water to the sample. Shake the stopper cylinder vigorously for at least for 30 seconds. Loosen the stopper of glass cylinder to release the vapor pressure in the cylinder but do not remove the stopper. Tighten the stopper and allow the stopper cylinder to sit undisturbed at ambient temperature upright on the leveled surface for a period of at least five minutes to allow the water-ethanol mixture to settle to the bottom.

E-6.2 If separation of two layers is not complete lightly tap the cylinder to encourage the complete separation. Record the level of aqueous layer in glass cylinder by reading the measurement at the lowest part of the meniscus.

> Presence of Ethanol percent in ethanol doped Motor Gasoline = Volume of aqueous layer observed in graduated cylinder – 30 ml (water added during testing).

E-6.3 Plot a graph on various values obtained above as ordinate (*X*-axis) against corresponding concentration of ethanol used in the standard blend as abscissa (*Y*-axis) including that of the observation made on the blank.

E-7 PROCEDURE

E-7.1 Take 100 ml sample of ethanol-blended Motor Gasoline under test in 150 ml graduated stoppered clean and dry glass cylinder. Add precisely 30 ml water to the sample taken in the glass cylinder and place the stopper tightly. Shake the stoppered cylinder vigorously for at least 30 seconds. Loosen the stopper of glass cylinder to release the vapor pressure in the cylinder but do not remove the stopper. Tighten the stopper and allow the stoppered cylinder to sit undisturbed at ambient temperature upright on the leveled surface for a period of at least five (5) minutes to allow the water-ethanol mixture to settle to the bottom.

E-7.2 If separation of two layers is not complete lightly tap the cylinder to encourage the complete separation. Record the level of aqueous layer in glass cylinder by reading the measurement at the lowest part of the meniscus.

> Presence of Ethanol percent, in ethanol doped Motor Gasoline = Volume of aqueous layer observed in graduated cylinder – 30 ml (water added during testing).

E-8 CALCULATION

Observation made under **E-7-2**, is read against the standard plot in **E-6.3** and the corresponding concentration of ethanol value is obtained. Whenever there is a change in source of Motor Gasoline, for better results, it is recommended to re-plot the graph as mentioned in **E-6.3**.

E-9 REPORT

Report the corrected results obtained as per **E-8** to the nearest 0.5 ml.

E-10 PRECISION AND BIAS

E-10.1 In the absence of calibration plot the value observed in aqueous phase can be calculated to the nearest percentage volume as per the under mentioned precision levels.

E-10.2 Based on the field test data, it was observed that a maximum of 9.0 ml (average) of ethanol content is extracted in to the aqueous phase as against 10 ml ethanol added in ethanol doped Motor Gasoline.

E-10.3 The standard deviation of the test results at 5 percent ethanol content by volume is ± 0.3 percent and for 10 percent ethanol content by volume is ± 0.50 percent.

E-10.4 Considering the precision of the graduated glass cylinder, the maximum possible measurement can be treated as 9.0 percent by volume with standard deviation of ±1.0 percent. Thus the repeatability under the method is as under:

Percentage	*Repeatability / Reproducibility*
5 percent (*v/v*)	0.5 ml (Approximately)
10 percent (*v/v*)	1.0 ml (Approximately)

(*Page* 22, *Annex* F) — Substitute the following for the existing Annex:

ANNEX F
(*Foreword*)

EURO IV/BHARAT STAGE IV EMMISSION NORMS COMPLIANT — SPECIFICATIONS FOR MOTOR GASOLINE (REQUIREMENTS)

Sl No.	*Characteristics*	*Requirements*		*Method of Test Ref to [P:] of IS 1448/ Annex of IS 2796/ISO/ Alternate Methods*
(1)	(2)	(3)	(4)	(5)
i)	Grade	MG 91	MG 95	
ii)	Colour, visual	As decided by Refiners/Marketers		—
iii)	Density @ 15°C, kg/m^3	720-775	720-775	[P : 16]1)/ISO 3675/ASTM D 4052
iv)	Distillation:			[P : 18]1)/ISO 3405/ASTM D 86
	a) Recovery up to 70°C (E-70), percent by volume	10-45	10-45	—
	b) Recovery up to 100°C (E-100), percent by volume	40-70	40-70	—
	c) Recovery up to 150°C (E-150), percent by volume, *Min*	75	75	—
	d) Final boiling point, °C, *Max*	210	210	—
	e) Residue, percent by volume, *Max*	2	2	—
v)	Research Octane Number (RON), *Min*	91	95	[P : 27]/ISO 5164/ASTM D 2699
vi)	Motor Octane Number (MON), *Min*	81	85	[P : 26]/ISO 5163/ASTM D 2700
vii)	Existent gum, g/m^3, *Max*	40	40	[P : 29]/ISO 6246/ASTM D 381
viii)	Sulphur, total, mg/kg, *Max*	50	50	[P : 34]1)/[P : 83]/ISO 4260/ISO 14596/ISO 20847/ASTM D 1266/ D 2622/D 3120/D 5453
ix)	Lead content (as Pb), g/1, *Max*	0.005	0.005	[P : 80]1)/IP 224/IP 352/ASTM D 5059
x)	Reid vapour pressure (RVP) at 38°C, kPa, *Max* (*see* Note 1)	60 (67)	60 (67)	[P : 39]1) /ISO 3007/ASTM D 323 (Wet method)/D 5191(Dry method)/ EN 13016/Annex A (Dry method) (*see* Note 2)
xi)	Vapour Lock Index (VLI), (VLI = 10 RVP + 7E 70), *Max* (*see* Notes 1& 2):			
	a) Summer (*see* Note 3)	750 (900)	750 (900)	—
	b) Other Months	950 (1 050)	950 (1 050)	—
xii)	Benzene content, percent by volume, *Max*	1	1	ASTM D 3606 (*see* Note 4)/ ASTM D 5580/D 6277/D 6730
xiii)	Copper Strip Corrosion (for 3 h @ 50°C), *Max*	Not more than 1	Not more than 1	[P : 15]/ISO 2160/ASTM D 130
xiv)	Water tolerance of gasoline-alcohol blends, temperature for phase separation, °C, *Max*:			Annex B
	a) Summer	10	10	
	b) Winter (*see* Note 5)	0	0	

Sl No.	*Characteristics*	*Requirements*		*Method of Test Ref to [P:] of IS 1448/ Annex of IS 2796/ISO/ Alternate Methods*
(1)	(2)	(3)	(4)	(5)
xv)	Engine intake system cleanliness (*see* Note 6)	Report-MFA Used	Report-MFA Used	—
xvi)	Olefin content, percent by volume, *Max*	21	18	[P : 23] 1)/ISO 3837/ASTM D 1319/ D 6730
xvii)	Oxidation stability, minutes, *Min*	360	360	[P : 28]/ISO 7536/ASTM D 525/IP 40
xviii)	Aromatic content, percent by volume, *Max*	35	35	[P : 23]/ISO 3837/ASTM D 1319/ D 6730/D 5580
xix)	Oxygen content, percent by mass, *Max*	2.7	2.7	EN 1601/IP 408/ASTM D 4815
xx)	Oxygenates, percent by volume, *Max*			ASTM D 4815/Annex C
	a) Methanol	Nil	Nil	
	b) Ethanol (*see* Note 7)	5	5	Annex E
	c) Iso-Propyl alcohol	10	10	
	d) Iso-butyl alcohol	10	10	
	e) Tertiary-butyl alcohol	7	7	
	f) Ethers containing 5 or more 'C' atoms per molecule	15	15	
	g) Other Oxygenates (*see* Note 8)	8	8	

NOTES

1 Limits given in ( ) shall be applicable to 5 percent (*v/v*) ethanol-blended gasoline only. Those areas where blending of ethanol in gasoline is compulsory the dosage of ethanol shall be 4.75 ± 0.25 percent (*v/v*).

2 For the gasoline-alcohol blends, the dry vapour test method given in Annex A shall be followed.

3 Summer shall be the period from May to July.

4 It is applicable only for non-alcoholic motor gasoline.

5 In winter it is expected that temperature may be lower than 0°C in the northern hilly region and hence phase separation shall not take place till -10°C.

6 Use of multifunctional additives (MFA) is a requirement for assuring adequate fuel system and intake system cleanliness performance in engines. Refiners/Marketers of motor gasoline have to ensure the MFA2) has proper credentials from internationally accepted test laboratories/authorities, of having passed a minimum of one of the tests in each of the two categories of deposit control performance indicated below:

	Deposit Type	*Test No.*	*Test Equipment*	*Test Method*	*Pass Limit (Average IVD (Weight)*
a)	Intake Valve Deposit (IVD)	1	BMW IVD Test	ASTM D 5500-97	100 mg/valve, *Max*
		2	MB M102E IVD	CEC F-05-A-93	50 mg/valve, *Max*
		3	FORD 2.3 Litre IVD	ASTM D 6201	90 mg/valve, *Max*
		4	MB M111-IVD Test	CEC F-20-A-98	50 mg/valve, *Max*
					Limit (% Injector Flow Loss)
					Pass Limit (% Injector Flow Loss)
b)	Port Fuel Injector Deposit (PFI)	1	Chrysler PFI Test	ASTM D 5598-95 A	5, *Max*
		2	PFI Deposit Rig	ASTM D 6421	10 percent, *Max*

Other performance tests may be added as and when they reach qualified/standard test status.

7 Stabilizing agents may be added. For determination of ethanol content ASTM D 5599 or ASTM D 4815 shall be employed. For routine analysis, Method ASTM D 5845 (FTIR) or/and Water extraction method may be employed (*see* Annex E).

8 Acetone is not permitted except when present as a by-product of the manufacture of certain oxygenate compounds and then only up to 0.8 percent (*v/v*).

9 All the test methods referred to in this standard include a precision statement. The Interpretation of results based on test method/Precision shall be used whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.

10 Type test for Phosphorus content in Motor Gasoline shall be introduced shortly.

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1) In case of dispute, this method shall be the referee method.

2) MFAs which are certified against National Generic Certification Option as per US EPA-97 final Rule (40 CFR Part 80 Certification Standards for Deposit Control Additives) can also be used by fuel refiners/Marketers at treatment levels not less than the Lower Additives Concentration (LAC) limits, as these MFAs meet the criteria for acceptance mentioned above.

(PCD 3)

Reprography Unit, BIS, New Delhi, India

Indian Standard
MOTOR GASOLINE — SPECIFICATION
(Fourth Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for two grades of unleaded motor gasoline under each of BS II and BS III categories complying with BS II and BS III emission norms suitable for use as a fuel in the automobile spark-ignition internal combustion engines of vehicles.

1.2 This standard also applies to blends of gasoline with organic oxygenates such as alcohols and ethers.

1.3 This standard does not purport to address all of the safety-problems associated with its use. It is the responsibility of the user of this specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling: Part 1 Manual sampling (*first revision*)
1448	Method of test for petroleum and its products:
[P: 10] : 1970	Cloud point and pour point (*first revision*)
[P: 15] : 2004/ ISO 2160 : 1998	Petroleum products corrosiveness to copper strip test (*third revision*)
[P : 16] : 1990	Density of crude petroleum and liquid petroleum products by hydrometer method (*third revision*)
[P : 18] : 1991	Distillation (*second revision*)
[P : 23] : 2004/ ISO 3837 : 1993	Liquid petroleum gases— Determination of hydrocarbon types fluorescent indicator absorption method (*fourth revision*)
[P : 26] : 1960	Knock characteristics of motor fuels by motor method
[P : 27] : 1960	Knock characteristics of motor fuels by research method
[P : 28] : 1985	Oxidation stability of motor gasoline and aviation fuels (*third revision*)
[P : 29] : 2004/ ISO 6246 : 1995	Petroleum products — Gum content of light and middle distillate fuels— Jet evaporation method (*third revision*)
[P : 30] : 1970	Sediment in crude and fuel oils by extraction (*first revision*)
[P : 34] : 1979	Determination of sulphur in petroleum products (lamp method) (*second revision*)
[P : 39] : 1967	Vapour pressure by Reid method (*first revision*)
[P : 80] : 1967	Determination of trace elements in petroleum products — Lead
[P : 82] : 1974	Total lead content in gasoline by iodine monochloride method
[P : 83] : 1974	Determination of sulphur by Wickbold oxyhydrogen method
[P : 147] : 1998	Determination of potential gum in motor gasoline
15464 : 2004	Anhydrous ethanol for use as automotive fuel

3 GRADES

There shall be two grades of motor gasoline under each of Bharat Stage II (BS II) and Bharat Stage III (BS III) categories as given below:

a) BS-II category for vehicles complying to BS II emission norms:
   1) MG 88 (Motor Gasoline with RON of 88); and
   2) MG 93(Motor Gasoline with RON of 93).

b) BS-III category for vehicles complying to BS III emission norms:
   1) MG 91(Motor Gasoline with RON of 91); and
   2) MG 95(Motor Gasoline with RON of 95).

4 REQUIREMENTS

4.1 General

The material shall be a refined petroleum distillate free from undissolved water, foreign matter and other visible impurities. It may also contain 5 percent (*v/v*), *Max* anhydrous denatured ethanol [99.5 percent (*v/v*)] conforming to IS 15464 apart from suitable additives (*see* **4.1.1** to **4.14**) in appropriate concentration to improve the quality of gasoline.

10 percent ethanol blended motor gasoline (E10) shall contain 10 percent (*v/v*), *Max* anhydrous denatured ethanol [99.5 percent (*v/v*)] conforming to IS 15464 apart from suitable additives (*see* **4.1.1** to **4.14**) in appropriate concentration to improve the quality of gasoline.

If organic oxygenates are used as blending components and/or stabilizing agents as specified in **4.2.1** the amount of such oxygenates shall comply with the limiting requirements as specified in **4.2.2.** No other oxygenates to be blended in 10 percent ethanol blended motor gasoline (E 10).

4.1.1 *Anti-oxidants*

Suitable antioxidants may be added in sufficient concentration to meet the requirements for oxidation stability as specified in Table 1. Some anti-oxidants of proven chemistry are listed below (this is not an exhaustive list):

a) N, N'-disecondary-butyl-paraphenylene diamine;
b) 2,4-dimethyl-6-tertiary butyl phenol;
c) 4-methyl-2, 6-ditertiary-butyl phenol;
d) N, N' – di-isopropyl-paraphenylene diamine;
e) N-Normal butyl-*p*-aminophenol; and
f) Mixture of N,N'-disecondary-butyl-paraphenylene diamine and disalicylidene-N-methyl dipropylene triamine.

Addition of anti-oxidants and MFA shall be mandatory for 10 percent ethanol blended motor gasoline (E 10).

4.1.2 *Metal Deactivators*

Suitable metal deactivators shall be added in sufficient concentration wherever required. Some metal deactivators of proven chemistry are listed below (this is not an exhaustive list):

a) N, N'-disalicylidene-propylene-diamine;
b) N,N'-disalicylidene-ethylene diamine; and
c) N,N'-disalicylidene-methylamine-dipropylene-triamine.

Suitable metal deactivators shall be added on sufficient concentration for 10 percent ethanol blended motor gasoline (E10).

4.1.3 *Dyes*

A suitable dye shall be added in such a proportion so as to satisfy the requirements for colour.

4.1.4 Gasoline multifunctional additives (MFA), other than those mentioned in **4.1.1** and **4.1.2** such as detergent/dispersant, rust, corrosion inhibitors and dehazer additives may be added in appropriate concentration to improve the quality of the fuel. Phosphorus containing additives shall not be used.

4.2 Organic Oxygenates

4.2.1 *Permitted Components*

The following components shall be used either singly or as mixtures as blending components or as stabilizing agents to prevent phase separation of the gasoline/ oxygenate components of the blend:

a) *Alcohols* — Ethanol, isopropyl alcohol (propane-2-ol), butyl alcohol (butane-1-ol), tertiary butyl alcohol (2-methyl-2-propanol), isobutyl alcohol (2-methyl-1-propanol) and other monoalcohols (excluding methanol) with a final boiling point not exceeding 215°C.
b) *Ethers* – Methyl tertiary butyl ether (MTBE), tertiary amyl methyl ether (methoxy-2-methyl butane) TAME, ethyl tertiary butyl ether (2-ethoxy-2-methyl propane) ETBE and other ethers (R-O-R) with final boiling point not exceeding 215°C and with molecules containing five or more carbon atoms.

4.2.2 *Content*

When tested, the content of the individual components listed in Table 1 shall comply with the limiting requirements as given in Table 1.

Mixtures of any of the individual components defined in **4.2.1** are permitted but the total oxygen content in the gasoline shall not exceed 2.7 percent by mass and the content of any of the individual components given at Sl No. (xxii) of Table 1 shall not exceed the corresponding limiting requirements.

4.3 Other Requirements

The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods referred in col 5 of the Table 1.

For 10 percent ethanol blended motor gasoline (E10), the material shall also comply with the requirements prescribed in Table 2 when tested according to the methods referred in col 5 of Table 2.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier and subject to the provisions of Red Tariff No. 18 Rules and Rates for the Conveyance by Rail of Explosives and other Dangerous Goods, issued by the Indian Railways Conference Association, with any alterations or additions made thereafter.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.2 Each container shall also be marked with the following information:

a) Name and grade of the material;
b) Indication of the source of manufacture, initials or trade-mark, if any;
c) Volume of the contents, in litres;
d) Year of manufacture or packing; and
e) The caution label 'FLAMMABLE' together with the corresponding symbols for labelling dangerous goods as given in Fig. 5 of IS 1260 (Part 1).

5.2.3 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

5.2.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

Table 1 Requirements for Motor Gasoline

(*Clauses* 4.1.1, 4.2.2 *and* 4.3)

Sl No.	**Characteristics**	**Requirements**				**Method of Test Ref to [P :] of IS 1448/Annex of IS 2796/ISO/ Alternate Methods**
		BS-II Category		BS-III Category		
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Grade	MG 88	MG 93	MG 91	MG 95	
ii)	Colour, visual	As decided by Refiners/ Marketers				—
iii)	Density @ 15 °C, kg/m^3	710-770	710-770	720-775	720-775	[P : 16]1)/ ISO 3675 / ISO 12185
iv)	Distillation :					
	a) Recovery up to 70 °C (E-70), percent by volume	10-45	10-45	10-45	10-45	
	b) Recovery up to 100 °C (E-100), percent by volume	40-70	40-70	40-70	40-70	
	c) Recovery up to 150 °C (E-150), percent by volume, *Min*	—	—	75	75	[P : 18]1) / ISO 3405
	d) Recovery up to 180 °C (E-180), percent by volume, *Min*	90	90	—	—	
	e) Final boiling point, °C, *Max*	215	215	210	210	
	f) Residue, percent by volume, *Max*	2	2	2	2	
v)	Research octane number (RON), *Min*	88	93	91	95	[P : 27] / ISO 5164 1)
vi)	Motor octane number (MON), *Min*	—	—	81	85	[P : 26] / ISO 5163 1)
vii)	Anti-knock index (AKI), *Min*	84	88	—	—	(RON+MON)/2
viii)	Existent gum, g/m^3, *Max*	40	40	40	40	[P : 29]1) / ISO 6246
ix)	Potential gum, g/m^3, *Max* (*see* Note 1)	50	50	—	—	[P:147] (*see* Note 1)
x)	Sulphur, total, percent by mass, *Max*	0.050	0.050	0.015	0.015	[P : 34]1) / ISO 4260, ISO 14596, ISO 20847 1)
xi)	Lead content (as Pb), g/l, *Max*	0.013	0.013	0.005	0.005	[P : 80]1)
xii)	Reid vapour pressure (RVP) at 38°C, kPa (*see* Note 2)	35-69 (35-67)	35-60 (35-67)	60 (67) *Max*	60 (67) *Max*	[P : 39]1)/ ISO 3007 (Wet method) Annex A (Dry method) (*see* Note 3)
xiii)	Vapour lock index (VLI), (VLI=10 RVP + 7E 70) *Max* (*see* Notes 2 and 3)					
	a) Summer (*see* Note 5)	750 (900)	750 (900)	750 (900)	750 (900)	
	b) Other months	950 (1 050)	950 (1 050)	950 (1 050)	950 (1 050)	

Table 1 (*Concluded*)

(1)	(2)	(3)	(4)	(5)	(6)	(7)
xiv)	Benzene content, percent by volume, *Max*	3	3	1	1	ASTM D 3606 (ASTM D 5580, (see Note 4)
xv)	Copper strip corrosion (for 3 h @ 50°C), *Max*	Not more than 1	Not more than 1	Not more than 1	Not more than 1	[P : 15][1)] / ISO 2160
xvi)	Water tolerance of gasoline-alcohol blends, temperature for phase separation, °C, *Max*					Annex B
	a) Summer	10	10	10	10	
	b) Winter (*see* Note 6)	0	0	0	0	
xvii)	Engine intake system cleanliness (*see* Note 7)	Report-MFA Used	Report-MFA Used	Report-MFA Used	Report-MFA Used	—
xviii)	Olefin content, percent by volume, *Max*	—	—	21	18	[P : 23][1)] / ISO 3837
xix)	Oxidation stability, *Min*	—	—	360	360	[P : 28][1)] / ISO 7536
xx)	Aromatic content, percent by volume, *Max*	—	—	42	42	[P : 23][1)] / ISO 3837
xxi)	Oxygen content, percent by mass, *Max*	2.7	2.7	2.7	2.7	Annex C and Annex D
xxii)	Oxygenates, percent by volume, *Max*					Annex C
	a) Methanol	Nil	Nil	Nil	Nil	
	b) Ethanol (*see* Note 8)	5	5	5	5	Annex E
	c) Iso-propyl alcohol	10	10	10	10	
	d) Iso-butyl alcohol	10	10	10	10	
	e) Tertiary-butyl alcohol	7	7	7	7	
	f) Ethers containing 5 or more 'C' atoms per molecules	15	15	15	15	
	g) Other oxygenates (*see* Note 9)	8	8	8	8	

NOTES

1 The test shall be carried out at the refinery end only on the gasoline before addition of multifunctional detergent/dispersants additives, as these may interfere with the test.

2 Limits given in ( ) shall be applicable to 5 percent *(v/v)* ethanol-blended gasoline only. Those areas where blending of ethanol in gasoline is compulsory the dosage of ethanol shall be 4.75 ± 0.25 *(v/v)*.

3 For the gasoline-alcohol blends, the dry vapour test method given in Annex A shall be followed.

4 It is applicable only for non-alcoholic motor gasoline.

5 Summer shall be the period from May to July.

6 In winter it is expected that temperature may be lower than 0°C in the northern hilly region and hence phase separation shall not take place till –10°C.

7 Use of multifunctional additives (MFA) is a requirement for assuring adequate fuel system and intake system cleanliness performance in engines. Refiners/Marketers of motor gasoline have to ensure the MFA[2)] has proper credentials from internationally accepted test laboratories/authorities, of having passed a minimum of one of the tests in each of the two categories of deposit control performance indicated below:

	Deposit Type	*Test No.*	*Test Equipment*	*Test Method*	*Pass Limit (Average IVD) (Weight)*
a)	Intake valve deposit (IVD)	1	BMW IVD Test	ASTM D 5500-97	100 mg/ valve, *Max*
		2	MB M102E IVD	CEC F-05-A-93	50 mg/ valve, *Max*
		3	FORD 2.3 Litre IVD	ASTM D 6201	90 mg/ valve, *Max*
		4	MB M111-IVD Test	CEC F-20-A-98	50 mg/ valve, *Max* Limit (percent injector flow loss) Pass limit (percent injector flow loss)
b)	Port fuel injector deposit (PFI)	1	Chysier PFI test	ASTM D 5598-95 A	5, *Max*
		2	PFI deposit rig	ASTM D 6421	10 percent, *Max*

Other performance tests may be added as and when they reach qualified/standard test status.

8 Stabilizing agents may be added. For determination of ethanol content ASTM D 5599 or ASTM D 4815 shall be employed. For routine analysis, method ASTM D 5845 (FTIR) or/and water extraction method may be employed (*see* Annex E).

9 Acetone is not permitted except when present as a by-product of the manufacture of certain oxygenate compounds and then only up to 0.8 percent *(v/v)*.

10 All the test methods referred to in this standard include a precision statement. The interpretation of results based on test method/precision shall be used whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.

[1)] In case of dispute, this method shall be the referee method.

[2)] MFAs which are certified against National Generic Certification Option as per US EPA-97 final rule (40 CFR Part 80 certification standards for deposit control additives) can also be used by fuel refiners/marketers at treatment levels not less than the Lower Additives Concentration (LAC) limits, as these MFAs meet the criteria for acceptance mentioned above.

Table 2 Requirements for 10 percent Ethanol Blended Motor Gasoline (E 10)

(*Clause* 4.3)

Sl No.	Characteristics	Requirements				Method of Test, Ref to [P :] of IS 1448/Annex of IS 2796/ISO/ Alternate Methods
		BS-II Category		BS-III Category		
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Grade	MG 88	MG 93	MG 91	MG 95	
ii)	Colour, visual	As decided by Refiners/Marketers				—
iii)	Density @ 15 °C, kg/m^3	710-770	710-770	720-775	720-775	[P : 16]1) / ISO 3675/ ISO 12185
iv)	Distillation:					
	a) Recovery up to 70°C (E-70), percent by volume	10-55 (summer) 10-58 (winter)	10-55 (summer) 10-58 (winter)	10-55 (summer) 10-58 (winter)	10-55 (summer) 10-58 (winter)	
	b) Recovery up to 100°C (E-100), percent by volume	40-70	40-70	40-70	40-70	
	c) Recovery up to 150°C (E-150), percent by volume, *Min*	—	—	75	75	[P : 18] / ISO 34051)
	d) Recovery up to 180°C (E-180), percent by volume, *Min*	90	90	—	—	
	e) Final boiling point, °C, *Max*	215	215	210	210	
	f) Residue, percent by volume, *Max*	2	2	2	2	
v)	Research octane number (RON), *Min*	88	93	91	95	[P : 27] / ISO 51641)
vi)	Motor octane number (MON), *Min*	—	—	81	85	[P : 26] / ISO 51631)
vii)	Anti-knock index (AKI), *Min*	84	88	—	—	(RON+MON)/2
viii)	Existent gum, g/m^3, *Max*	40	40	40	40	[P : 29]1) / ISO 6246
ix)	Potential gum, g/m^3, *Max*	50	50	—	—	[P : 147] (*see* Note 1)
x)	Sulphur, total, percent by mass, *Max*	0.050	0.050	0.015	0.015	[P : 34]1)/ ISO 4260, ISO 14596, ISO 208471)
xi)	Lead content (as Pb), g/l, *Max*	0.013	0.013	0.005	0.005	[P : 80]1)
xii)	Reid vapour pressure (RVP) at 38°C, kPa	35-67	35-67	67, *Max*	67, *Max*	[P : 39]1)/ ISO 3007 (Wet method) Annex A (Dry method) (*see* Note 3)
xiii)	Vapour lock index (VLI), (VLI=10 RVP + 7E 70) *Max* (*see* Note 3)					
	a) Summer (*see* Note 4)	1 050	1 050	1 050	1 050	
	b) Other months	1 100	1 100	1 100	1 100	
xiv)	Benzene content, percent by volume, *Max*	3	3	1	1	ASTM D 5580
xv)	Copper strip corrosion (for 3 h @ 50°C), *Max*	Not more than 1	Not more than 1	Not more than 1	Not more than 1	[P : 15]1) / ISO 2160
xvi)	Water tolerance of gasoline-alcohol blends, temperature for phase separation, °C, *Max*					Annex B
	a) Summer	10	10	10	10	
	b) Winter (*see* Note 5)	0	0	0	0	
xvii)	Engine intake system cleanliness (*see* Note 6)	Report-MFA Used	Report-MFA Used	Report-MFA Used	Report-MFA Used	—
xviii)	Olefin content, percent by volume, *Max*	—	—	21	18	[P : 23]1) / ISO 3837
xix)	Oxidation stability, *Min*	—	—	360	360	[P : 28]1) / ISO 7536
xx)	Aromatic content, percent by volume, *Max*	—	—	42	42	[P : 23]1) / ISO 3837
xxi)	Oxygen content, percent by mass, *Max*	3.5	3.5	3.5	3.5	Annex C and Annex D
xxii)	Oxygenates, percent by volume Ethanol (*see* Notes 2 and 7)	10	10	10	10	Annex C

NOTES

1 The test shall be carried out at the refinery end only on the gasoline before addition of multifunctional detergent/dispersants additives, as these may interfere with the test.

2 The dosage of ethanol shall be 9.75 ± 0.25 percent (*v/v*).

3 For the gasoline-alcohol blends, the dry vapour test method given in Annex A shall be followed.

Table 2 (*Concluded*)

4 Summer shall be the period from May to July.

5 In winter it is expected that temperature may be lower than 0°C in the northern hilly region and hence phase separation shall not take place till −10°C.

6 Use of multifunctional additives (MFA) is a requirement for assuring adequate fuel system and intake system cleanliness performance in engines. Refiners/Marketers of motor gasoline have to ensure the MFA[2] has proper credentials from internationally accepted test laboratories/authorities, of having passed a minimum of one of the tests in each of the two categories of deposit control performance indicated below:

	Deposit Type	*Test No.*	*Test Equipment*	*Test Method*	*Pass Limit (Average IVD (Weight)*
a)	Intake valve deposit (IVD)	1	BMW IVD Test	ASTM D 5500-97	100 mg/valve, *Max*
		2	MB M102E IVD	CEC F-05-A-93	50 mg/valve, *Max*
		3	FORD 2.3 Litre IVD	ASTM D 6201	90 mg/valve, *Max*
		4	MB M111-IVD Test	CEC F-20-A-98	50 mg/valve, *Max*
					Limit (percent injector flow loss)
					Pass limit (percent injector flow loss)
b)	Port fuel injector deposit (PFI)	1	Chrysier PFI test	ASTM D 5598-95 A	5, *Max*
		2	PFI deposit rig	ASTM D 6421	10 percent, *Max*

7 Stabilizing agents shall be added. For determination of ethanol content ASTM D 5599 or ASTM D 4815 shall be employed. For routine analysis, method ASTM D 5845 (FTIR) may be employed (*see* Annex E).

8 Acetone is not permitted except when present as a by-product of the manufacture of certain oxygenate compounds and then only up to 0.8 percent (*v/v*).

9 All the test methods referred to in this standard include a precision statement. The interpretation of results based on test method/precision shall be used whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.

10 There shall be proper labelling of E10 (10 percent ethanol blended motor gasoline) fuel at the retail outlets.

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[1] In case of dispute, this method shall be the referee method.

[2] MFAs which are certified against National Generic Certification Option as per US EPA-97 final rule (40 CFR Part 80 certification standards for deposit control additives) can also be used by fuel refiners/marketers at treatment levels not less than the Lower Additives Concentration (LAC) limits, as these MFAs meet the criteria for acceptance mentioned above.

ANNEX A

[*Table* 1, *Sl No.* (xii) and *Table* 2, *Sl No.* (xii)]

TEST METHOD FOR VAPOUR PRESSURE OF GASOLINE-ALCOHOL BLEND (DRY METHOD)

A-1 GENERAL

This test method covers the determination of the absolute vapour pressure of gasoline and gasoline-oxygenate blends.

NOTE — Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the 'vapour pressure' is an absolute pressure at 38°C in kilopascals. This vapour pressure differs from the true pressure of the sample due to some small sample vaporization and the presence of air in the confined space.

A-2 SUMMARY OF TEST METHOD

The fuel chamber of the vapour pressure apparatus is filled with the chilled sample and connected to the air chamber at 38°C. The apparatus is immersed in a bath at 38°C and is shaken periodically until a constant pressure is observed on the gauge attached to the apparatus. The gauge reading, suitably corrected, is reported as the vapour pressure.

A-3 SIGNIFICANCE AND USE

The method IS 1448 [P : 39] cannot be used to determine the vapour pressure of gasoline-oxygenate blends which contain water-extractable oxygenates because the fuel sample comes into contact with water. This test method is a modification of IS 1448 [P : 39] where contact with water has been eliminated.

A-4 APPARATUS

The construction of the required apparatus is described in **3** of IS 1448 [P : 39].

A-5 REAGENTS

A-5.1 Purity of Reagents

Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the relevant Indian Standard where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A-5.2 Acetone

WARNING — Extremely flammable. Irritating to skin, eyes and mucous membranes.

A-5.3 Naphtha

NOTE — Naphtha [Tag closed cup flash point below -6°C].

WARNING — Extremely flammable. Harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard.

A-6 HANDLING OF SAMPLES

A-6.1 The extreme sensitivity of vapour pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples. The provisions of this section apply to all samples for vapour pressure determinations.

A-6.2 Sample in accordance with IS 1447 (Part 1) except that water displacement must not be used.

A-6.3 Sample Container Size

The size of the sample container from which the vapour pressure sample is taken as 1 litre. It will be 70 to 80 percent filled with the sample.

A-6.4 Precautions

A-6.4.1 Determine vapour pressure as the first test run on a sample. Do not withdraw more than one sample from the sample container for this test.

A-6.4.2 Protect samples from excessive heat prior to testing.

A-6.4.3 Do not test samples in leaky containers. Discard them and obtain new samples.

A-6.4.4 Discard samples that have separated into two phases and obtain new samples (*see* Note 2 under **A-8.3**).

A-6.5 Sample Handling Temperature

In all case, cool the sample container and contents to 0 to 1°C before the container is opened. To ensure sufficient time to reach this temperature, directly measure the temperature of a similar liquid in a like container placed in the cooling bath at the same time as the sample.

A-7 PREPARATION FOR TEST

A-7.1 Verification of Sample Container Filling

With the sample at a temperature of 0 to 1°C, take the container from the cooling bath, wipe dry with an absorbent material, unseal it, and examine its ullage. The sample content, as determined by use of a suitable gauge, must be equal to 70 to 80 percent of the container capacity.

A-7.1.1 Discard the sample if its volume is less than 70 percent of the container capacity.

A-7.1.2 If the container is more than 80 percent full, pour out enough sample to bring the container contents within the 70 to 80 percent range. Under no circumstances, may any sample poured out, be returned to the container.

A-7.2 Air Saturation of Sample in Sample Container

A-7.2.1 With the sample again at a temperature of 0 to 1°C take the container from the cooling bath, wipe it dry with an absorbent material, unseal it momentarily, taking care to prevent water entry, reseal it, and shake it vigorously. Return it to the bath for a minimum of 2 min.

A-7.2.2 Repeat **A-7.2.1** twice more. Return the sample to the bath and keep it there until the beginning of the procedure (*see* **A-8**).

A-7.3 Preparation of Fuel Chamber

Observe the apparatus preparation procedure of **A-8.5**, then store the stoppered fuel chamber and the sample transfer connection in a refrigerator or ice-water bath for a sufficient time to allow the chamber and the connection to reach a temperature of 0 to 1°C. If and ice-water bath is used, keep the chamber upright and not immersed over the top of the coupling threads. The transfer connection is inserted over the top of the coupling threads. The transfer connection is inserted into a plastic bag to keep it completely dry during cooling.

A-7.4 Preparation of Air Chamber

Observe the apparatus preparation procedure of **A-8.5**. Connect the gauge to the air chamber and close the lower opening securely with a dry No.6 rubber stopper. Make sure the stopper is inserted far enough to securely close the vent hole in the air chamber connection. Immerse the air chamber to at least 25 mm above its top in the water bath until the fuel chamber has been filled with the sample as described in **A-8.1**.

A-8 PROCEDURE

A-8.1 Sample Transfer

With everything in readings, remove the chilled sample container from the bath, dry it with absorbent material, uncap it, and dry and insert the chilled transfer apparatus (*see* Fig.1). Quickly place the chilled fuel chamber, in an inverted position, over the sample delivery tube of the transfer apparatus. Invert the entire system rapidly so that the fuel chamber is upright, with the end of the delivery tube touching the bottom of the fuel chamber. Fill the fuel chamber to the overflowing. Withdraw the delivery tube from the fuel chamber while allowing the sample to continue flowing up to the moment of complete withdrawal.

A-8.1.1 *Caution*

Make provision for suitable collection and disposal of the overflowing fuel to avoid fire hazard.

A-8.2 Assembly of Apparatus

Immediately remove the air chamber from the water bath and immediately dry the exterior of the chamber with absorbent material giving particular care to the connection between the air chamber and the fuel chamber. Remove the stopper after drying and immediately couple the two chambers. Not more than 10s shall be consumed in coupling the two chambers.

NOTE — When the air chamber is removed from the water bath is dried, and the stopper is removed, connect it to the fuel chamber without undue movements through the air, which could promote exchange of room temperature air with the 38°C air in the chamber.

A-8.3 Introduction of Apparatus into Bath

Turn the assembled vapour pressure apparatus upside down to allow the sample in the fuel chamber to run into the air chamber. With the apparatus still inverted, shake it vigorously eight times in a direction parallel to the length of the apparatus. With the gauge end up, immerse the assembled apparatus in the bath, maintained at 38 ± 0.1°C in an inclined position so that the connection of the fuel and air chambers is below the water level and may be carefully examined for leaks 25 mm above the top of the air chamber. Observe the apparatus for leakage throughout the test. Discard the test at any time a leak is detected.

NOTES

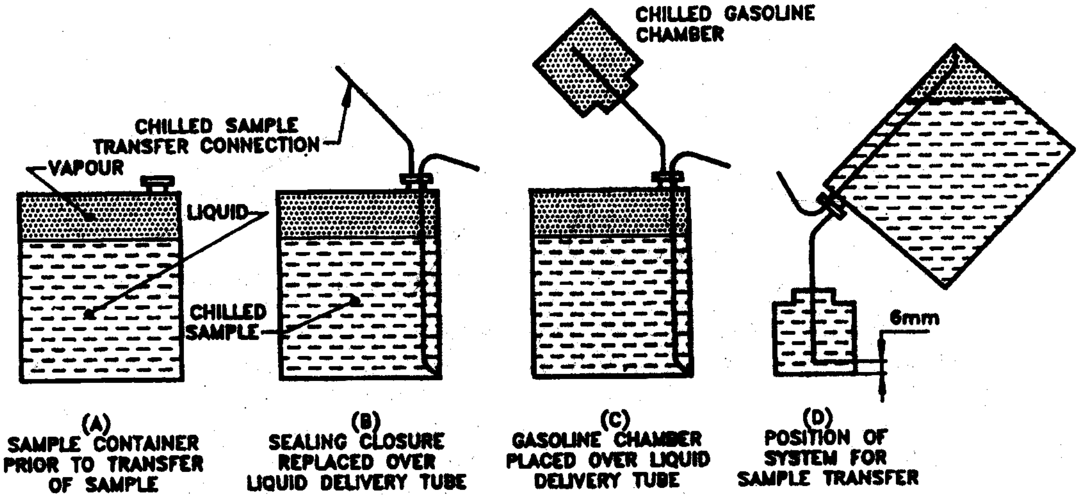
1 Liquid leaks are more difficult to detect than vapour leaks, and because the coupling between the chambers is normally in the liquid section of the apparatus give the coupling particular attention.

2 After the apparatus has been immersed in the bath check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer (*see* **A-8.1**). If the sample is contained in a non-transparent container, shake the sample vigorously for 5 s and then immediately pour a portion of the remaining sample into a clear glass container. Immediately after shaking this sample again for 5 s, observe the sample for phase separation. If this sample is not clear and bright, and free of a second phase, discard the test and the sample.

A-8.4 Measurement of Vapour Pressure

After the assembled vapour pressure apparatus has been immersed in the bath for at least 5 min, tap the pressure gauge lightly and observe the reading. Withdraw the apparatus from the bath and repeat **A-8.3**.

At intervals of not less than 2 min, perform **A-8.3**



(A) SAMPLE CONTAINER PRIOR TO TRANSFER OF SAMPLE

(B) SEALING CLOSURE REPLACED OVER LIQUID DELIVERY TUBE

(C) GASOLINE CHAMBER PLACED OVER LIQUID DELIVERY TUBE

(D) POSITION OF SYSTEM FOR SAMPLE TRANSFER

FIG. 1 SIMPLIFIED SKETCHES OUTLINING MELTHOD OF TRANSFERING SAMPLES TO GASOLINE CHAMBER FROM OPEN-TYPE CONTAINERS

until a total of not less than five shakings and gauge readings have been made continue thereafter, if necessary, until the last two consecutive gauge readings are constant, indicating equilibrium attainment. These operations normally require 20 to 30 min. Read the final gauge pressure to the nearest 0.25 kPa for gauges with intermediate graduations of 0.5 kPa or less than to the nearest 0.5 kPa for gauges with graduations of 1.0 to 2.5 kPa and record the values as the 'uncorrected vapour pressure' of the sample. Without undue delay remove the pressure gauge and, without attempting to remove any liquid which may be trapped in the gauge, check its reading against that of the manometer while both are subjected to a common steady pressure which is no more than 1.0 kPa different from the recorded 'uncorrected vapour pressure'. If a difference is observed between the gauge and manometer readings, the difference shall be added to or subtracted from the 'uncorrected vapour pressure' recorded for the sample being tested, and the resulting value shall be recorded as the vapour pressure of the sample.

NOTES

1 Cooling the assembly prior disconnecting the gauge will facilitate disassembly and reduce the amount of hydrocarbon vapour released into the room.

2 *Verification of sample integrity* — Disconnect the air chamber from the fuel chamber. Drain the sample from the air and fuel chambers as completely as possible into a dry (8-oz) 240 ml clear glass bottle. Seal the bottle and shake it vigorously for 5 s. If the sample is clear and bright and free of a second phase, note this observation and record the test is valid. If the sample is not clear and bright and free of second phase. Immerse the bottle in the 38°C water bath up to about 25 mm above the top of the sample level for 15 min in order to heat the sample to the test temperature. Remove the sample from the water bath and immediately shake it vigorously for 5 s and observe the sample. If the sample is not clear and bright and free of second phase, note this observation and record that the test is not valid because of phase separation. A fuel that is not clear and bright and free of second phase at this point of the test indicates that the fuel was contacted with sufficient water to exceed the water tolerance of the fuel during the test procedure. Water can most likely get into the test chambers during preparation of the fuel and air chambers (*see* **A-7.3** and **A-7.4**) or assembly of the air and fuel chambers (*see* **A-8.2**), or both, specially and fuel chambers (*see* **A-8.2**), or both specially if water baths are used for these procedures.

A-8.5 Preparation of Apparatus for Next Test

Thoroughly purge the air chamber of residual sample by filling it with warm water above 32°C and allowing it to drain. Repeat the purging at least five times. After disconnecting the pressure gauge from its manifold connection with the manometer, remove trapped centrifugal thrusts. This may be accomplished in the following manner, hold the gauge between the palms of the hands with the right hand on the face side and the threaded connection of the gauge forward. Extend the arms forward and upward at an angle of 45°, with the coupling of the gauge pointing in the same direction. Swing the arms downward through an arc of about 135° so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gauge by directing a small jet of air into its Bourdon tube for at least 5 min. Rinse both chambers and the sample transfer connection several times with petroleum naphtha, then several times with acetone, then blow dry using dried air. Stopper the fuel chamber and place it in the refrigerator or ice-water bath for the next test.

NOTE — If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top opening of the chamber closed as they pass through the water surface.

A-9 PRECAUTIONS

A-9.1 Gross errors can be obtained in vapour pressure measurements, if the prescribed procedure is not followed carefully. The following list emphasizes the importance of strict adherence to the precautions given in the procedure.

A-9.1.1 *Checking the Pressure Gauge*

Check all gauges against a manometer after each test in order to ensure high precision of results (*see* **A-8.4**). Read all gauges while the gauges are in a vertical position and after tapping them lightly.

A-9.1.2 Shake the container vigorously to ensure equilibrium of the sample with the air in the container (*see* **A-7.2**).

A-9.1.3 *Checking for Leaks*

Check the apparatus before and during each test for both liquid and vapour leaks.

A-9.1.4 *Sampling*

Because initial sampling and the handling of samples will greatly affect the final results, employ the utmost precaution and the most meticulous care to avoid losses through evaporation and even slight changes in composition (*see* **A-6.5** and **A-8.1**). In no case shall any part of the apparatus itself be used as the sample container prior to actually conducting the test.

A-9.1.5 *Purging the Apparatus*

Thoroughly purge the pressure gauge, the fuel chamber and the air chamber to be sure they are free of residual sample. [This is most conveniently done at the end of the previous test (*see* **A-8.5**)]. It is important to remove all water from the apparatus before cooling the gasoline chambers and heating the air chamber. In high-humidity conditions be alert for and avoid condensation on the transfer connection and interior walls of the apparatus.

A-10 REPORT

A-10.1 Reporting Results

Report to the nearest 0.25 kPa or 0.5 kPa the gauge result observed in **A-8.4**, after correcting for any difference between the gauge and manometer, as the vapour pressure in kilopascals without reference to temperature.

A-11 PRECISION AND BIAS

A-11.1 Precision

The following criteria should be used for judging the acceptability of results (95 percent confidence).

A-11.1.1 *Repeatability*

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Component Gauge Range	*Repeatability* kPa
0 to 100 kPa	4.90

A-11.1.2 *Reproducibility*

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

Component Gauge Range	*Reproducibility* kPa
0 to 100 kPa	7.790

A-11.2 Bias

There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

ANNEX B

[*Table* 1, *Sl No.* (xvi) and *Table* 2, *Sl No.* (xvi)]

TEST METHOD FOR WATER TOLERANCE (PHASE SEPARATION) OF GASOLINE-ALCOHOL BLEND

B-1 GENERAL

This test method determines the ability of gasoline-oxygenate blends to retain water in solution or in a stable suspension at the lowest temperature to which they are likely to be exposed in use.

B-2 SUMMARY OF TEST METHOD

The sample of fuel is cooled at a controlled rate to its expected use temperature and is periodically observed for both haze and phase separation. The apparatus as given in **4** of IS 1448 [P : 10] or a dry ice-isopropyl alcohol bath may be used. A maximum cooling rate of 2°C/mm is specified because phase separation in gasoline-oxygenate blends has a relatively long but unpredictable induction period.

B-3 SIGNIFICANCE AND USE

B-3.1 Some oxygenate-containing fuels, and gasoline-alcohol blends in particular have a very limited ability to retain water in solution or in stable suspension, and if the amount of water in the blend exceeds this limit, the fuel will separate into a lower oxygenate-rich aqueous phase and an upper oxygenate-lean hydrocarbon phase. The most important factor governing the ability of a specific fuel to retain water without such separation is its temperature. This method is intended to determine the maximum temperature at which the fuel will separate. The temperatures represent the maximum temperatures above which the fuel must not separate into two distinct phases.

B-3.2 Note that in tyhis test, actual separation of the sample into two distinct phases is the criterion for failure. The following are indications of phase separation.

B-3.2.1 The formation of droplets large enough to be detected by the unaided eye. They may be either clinging to sides of the container or collect on the bottom.

B-3.2.2 The formation of two layers separated by either common boundary, or a layer of emulsion. Formation of haze without one of these indications of separation is not cause for rejection.

B-4 APPARATUS

B-4.1 Test Container — Any glass container of about 100 ml capacity may be used. This container may be marked at the level of 40 ml.

B-4.2 Thermometers — Meeting the requirements of Specification E-1,ASTM thermometer 6°C (range -80 to +20°C, 1°C graduations, 76 mm immersion. Thermometer 6°F is the Fahrenheit equivalent of 6°C), spans the necessary range of temperatures. A thermometer must be provided for each container, mounted to pass through the stopper.

B-4.3 Viton Rubber Stopper — To fit sample container bored centrally for the test thermometer.

B-4.4 Cooling Bath — May be of similar dimensions to those specified in **4** of IS 1448 [P : 10] and provided with a jacket disk and gasket (as specified in the apparatus section) filled with an equal-volume mixture of water and 'Permanent' anti-freeze and provided with refrigeration coils capable of reducing its temperature to -40°C. Alternatively, a dry ice-isopropyl alcohol bath may be used.

B-5 SAMPLING AND HANDLING

B-5.1 Draw samples in accordance with IS 1447 (Part 1), except water displacement shall not be used.

B-5.2 Draw the samples in steel cans that have been solvent washed.

B-5.3 Store the samples in refrigerator (2 to 7°C) whenever not actually transferring sample.

B-5.4 Because gasoline-alcohol blends are hygroscopic as well as volatile, minimize contact with the atmosphere by keeping sample containers tightly closed except when transferring sample.

B-6 PROCEDURE

B-6.1 Warm the sample to 15°C and shake, to redissolve any water that may have settled out at the refrigerator temperature.

B-6.2 Cool the test container to 10 to 15°C. Carry out steps **B-6.3** through **B-6.5** as promptly as possible to minimize vaporization losses and absorption of water from the atmosphere.

B-6.3 Rinse out the cooled test container with some of the sample to be tested. Drain.

B-6.4 Pour about 40 ml of the sample into the test container. The precise amount is not critical, but it must be enough to submerge the thermometer bulb adequately, without being so much as to require an excessive amount of cooling time. If the sample has separated, as defined in **B-3.2**, terminate the test.

B-6.5 Seal the test container with the rubber stopper. Locate the thermometer bulb approximately at the centre of the fuel sample.

B-6.6 Cool the sample by intermittent immersion in or circulation of the coolant. The sample is not to be swirled or shaken while in the cooling bath. Starting at a cooling bath temperature not higher than 10°C or 16°C above the test temperature, cool the sample at a maximum rate of 2°C/min until phase separation occurs, or the test temperature is reached.

B-6.7 At 2°C intervals, remove the test container from the cooling bath and shake vigorously for 5 to 10s. Wipe the exterior of the sample container with a towel moistened with isopropyl alcohol to remove any condensation, and observe the condition of the sample for no more than 5s against a light coloured illuminated background.

B-6.8 It is likely that the sample will get hazy prior to actual phase separation as defined in **B-3.2**. Record the sample temperature at the first indication of haze (when cooling); and the temperature when the haze disappears (warming).

B-6.9 Record the temperature of phase separation (*see* **B-3.2**). Then allow the sample to warm at ambient temperature. Shake the sample vigorously after a temperature rise of 2°C, and observe. Record a 'warming' phase recombination temperature, and the haze disappearance temperature. Average these 'cooling' and 'warming' temperatures to determine the actual phase separation and haze point temperatures. Repeat this process for improved accuracy.

B-7 REPORT

B-7.1 Report the averaged haze point and phase separation temperatures found in **B-6.9**.

ANNEX C

[*Table* 1, *Sl No.* (xxi), (xxii) and *Table* 2, *Sl No.* (xxi), (xxii)]

DETERMINATION OF MTBE, ETBE, TAME, DIPE, TERTIARY-AMYL ALCOHOL AND C1 TO C4 ALCOHOLS IN GASOLINE BY GAS CHROMATOGRAPHY

C-1 GENERAL

C-1.1 This test method stipulates a procedure for determination of alcohols (methanol, ethanol, isopropanol, *n*-propanol, isobutanol, sec-butanol, tert-butanol, *n*-butanol, tert-pentanol) and ethers (MTBE, ETBE, TAME, DIPE) in gasoline by gas chromatography.

C-1.2 Individual alcohol are determined from 0.1 to 12.0 mass percent and individual ethers are determined from 0.1 to 20.0 mass percent. Equation used to convert to mass percent oxygen and to volume percent of individual compounds are available.

C-2 TERMINOLOGY

C-2.1 Low Volume Connector — A special union for connecting two lengths of tubing 1.6 mm inside diameter and smaller. Sometimes this is referred to as a zero dead volume union.

C-2.2 MTBE — methyl tertiary-butyl ether.

C-2.3 ETBE — ethyl tertiary-butyl ether.

C-2.4 TAME — tertiary-amyl methyl ether.

C-2.5 DIPE — diisopropyl ether.

C-2.6 Tertiary — **Amyl Alcohol** — tertiary-pentanol.

C-2.7 Oxygenates — Any oxygen-containing organic compounds which can be used as a fuel or fuel supplement, for example various alcohols and ethers.

C-2.8 Split Ratio — A term used in gas chromatography using capillary columns. The split ratio is the ratio of the total flow of the carrier gas to the sample inlet *versus* the flow of carrier gas to the capillary column expressed by:

$$\text{Split ratio} = (S + C)/C$$

where

S = flow rate at the splitter vent, and

C = flow rate at the column outlet.

C-2.9 TCEP — 1,2,3-tris-2-cyanoethoxypropane-a gas chromatographic liquid phase.

C-2.10 WCOT — A type of capillary gas chromatographic column prepared by coating the inside of the capillary with a thin film of stationary phase.

C-3 SUMMARY OF TEST METHOD

An appropriate internal standard such as 1,2-dimethoxy ethane (ethylene glycol dimethyl ether) is added to the sample which is then introduced into a gas chromatograph equipped with two columns and a column switching valve. The sample first passes into a polar TCEP column which elutes lighter hydrocarbons to vent and retains the oxygenated and heavier hydrocarbons.

After methylcyclopentane, but before DIPE and MTBE elute from the polar column, the valve is switched to backflush the oxygenates onto a WCOT non-polar column. The alcohols and ethers elute from the non-polar column in boiling point order, before elution of any major hydrocarbon constituents.

After TAME and benzene elute from the non-polar column, the column switching valve is switched back to its original position to backflush the heavy hydrocarbons.

The eluted components are detected by a flame ionization or thermal conductivity detector. The detector response, proportional to the component concentration, is recorded; the peak areas are measured; and the concentration of each component is calculated with reference to the internal standard.

C-4 SIGNIFICANCE AND USE

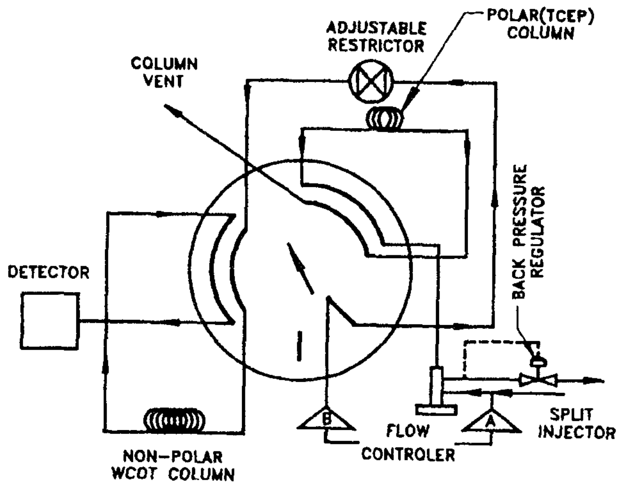
C-4.1 Ethers, alcohols and other oxygenates may be added to gasoline to increase the octane number and to reduce emissions. Type and concentration of various oxygenates are specified and regulated to ensure acceptable commercial gasoline quality. Drivability vapour pressure, phase separation exhaust and evaporative emissions are some of the concerns associated with oxygenated fuels.

C-4.2 This test method is applicable to both quality control in the production of gasoline and for the determination of deliberate or extraneous oxygenate additions or contamination.

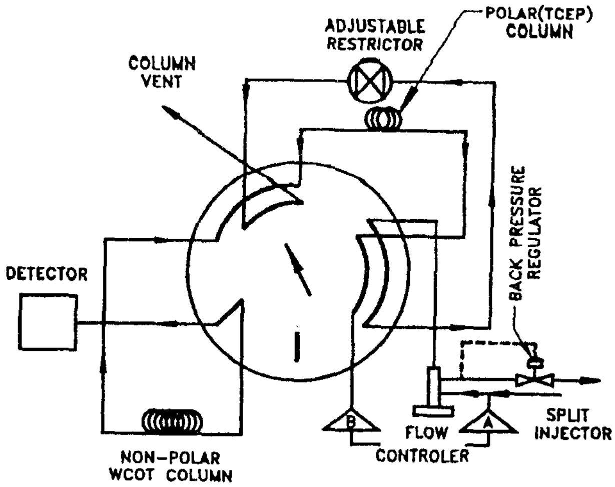
C-5 APPARATUS

C-5.1 Chromatograph

C-5.1.1 While any gas chromatographic system, which is capable of adequately resolving the individual ethers and alcohols, can be used for these analyses, a gas chromatographic instrument which can be operated under the conditions given in Table 3 and having a column switching and back flushing system equivalent to Fig. 2 has been found acceptable. Carrier gas flow controllers shall be capable of precise control where



VALVE IN RESET POSITION



VALVE IN BACKFLUSH POSITION

FIG. 2 ANALYSIS OF OXYGENATES IN GASOLINE SCHEMATIC DIAGRAM/REPRESENTATION OF CHROMATOGRAPHIC SYSTEM

Table 3 Chromatographic Operating Conditions

(*Clauses* C-5.1.1 *and* C-10.2)

Sl No. (1)	Temperatures (2)		Flows, ml/min (3)		Carrier Gas : Helium (4)	
i)	Column oven, °C	60	To injector	75	Sample size, microlitre	1.0-3.0
ii)	Injector, °C	200	Column	5	Split ratio	15 : 1
iii)	Detector					
	a) TCD, °C	200	Auxillary	3	Backflush, *Min*	0.2-0.3
	b) FID, °C	250	Makeup	18	Valve reset time	8-10 min
iv)	Valve, °C	60			Total analysis time	18-20 min

the required flow rates are low (*see* Table 3). Pressure control devices and gauges shall be capable of precise control for the typical pressures required.

C-5.2 Detector

A thermal conductivity detector or flame ionization detector, may be used. The system shall have sufficient sensitivity and stability to obtain a recorder deflection of at least 2 mm at a signal-to-noise ratio of at least 5 to 1 for 0.005 volume percent concentration of an oxygenate.

C-5.3 Switching and Back flushing Valve

A valve, to be located within the gas chromatographic column oven, capable of performing the functions described in **C-10** and illustrated in Fig. 2. The valve shall be of low volume design and not contribute significantly to chromatographic deterioration.

C-5.3.1 *Valco Model No. A 4C10WP, 1.6 mm Fittings*

This particular valve has been used in the majority of the analyses used for the development of **C-14**.

C-5.3.2 *Valco Model No. C10W, 0.8 mm Fittings*

This valve is recommended for use with columns of 0.32 mm inside diameter and smaller.

C-5.4 An automatic valve switching device shall be used to ensure repeatable switching times. Such a device shall be synchronized with injection and data collection times.

C-5.5 Injection System

The chromatograph should be equipped with a splitting-type inlet device if capillary column or flame ionization detection is used. Split injection is necessary to maintain the actual chromatographed sample size within the limits of column and detector optimum efficiency and linearity.

C-5.6 Sample Injectors

Any system capable of introducing a representative sample into the split inlet device. Microlitre syringes, automatic syringe injectors, and liquid sampling valves may be used.

C-5.7 Data Presentation or Calculation or Both

C-5.7.1 *Recorder*

A recording potentiometer or equivalent with a full scale deflection of 5 mV or less. Full-scale response time should be 1 s or less with sufficient sensitivity and stability to meet the requirements of **C-5.2**.

C-5.7.2 *Integrator or Computer*

Devices capable of meeting the requirements of **C-5.2** and providing graphic and digital presentation of the chromatographic data are recommended for use. Means shall be provided for determining the detector response. Peak heights or areas can be measured by computer, electronic integration or manual techniques.

C-5.8 Columns

C-5.8.1 *Polar Column*

This column performs a preparation of the oxygenates from volatile hydrocarbons in the same boiling point range. The oxygenates and remaining hydrocarbons are back flushed onto the non-polar column in **C-5.8.2**. Any column with equivalent or better chromatographic efficiency and selectivity to that described in **C-5.8.1.1** can be used. The column shall perform at the same temperature as required for the column in **C-5.8.2**.

C-5.8.1.1 *TCEP micro-packed column*

560 mm by 1.6 mm outside diameter by 0.38 mm inside diameter stainless steel tube packed with 0.14 to 0.15 g of 20 percent (mass/mass) TCEP on 80/100 mesh chromosorb P (AW). This column was used in the cooperative study to provide the precision and bias data referred to in **C-14**.

C-5.8.2 *Non-polar (Analytical) Column*

Any column with equivalent or better chromatographic efficiency and selectivity to that described in **C-5.8.2.1** and illustrated in Fig. 3 can be used.

C-5.8.2.1 *WCOT methyl silicon column*

30 m long by 0.53 mm inside diameter, fused silica WCOT column with a 2.6 μm film thickness of cross-linked methyl siloxane. This column was used in the operative study to provide the precision and bias data referred to in **C-14**.

C-6 REAGENTS AND MATERIALS

C-6.1 Carrier Gas — Carrier gas appropriate to the type of detector used. Helium has been used successfully. The minimum purity of the carrier gas used shall be 99.95 mol percent.

C-6.2 Standards for Calibration and Identification — Standards of all components to be analyzed and the internal standards are required for establishing identification by retention time as well as calibration for quantitative measurements. These materials shall be of known purity and free of the other components to be analyzed.

WARNING — These materials are flammable and harmful or fatal if ingested or inhaled.

C-6.3 Methylene Chloride

Used for column preparation. Reagent grade shall be free of non-volatile residue.

WARNING — Harmful if inhaled. High concentration may cause unconsciousness or death.

C-7 PREPARATION OF COLUMN PACKINGS

C-7.1 TCEP Column Packing

Any satisfactory method, used in the practice of the art that will produce a column capable of retaining the C_1 to C_4 alcohols and MTBE, ETBE, DIPE and TAME from components of the same boiling point range in a gasoline sample. The following procedure has been used successfully:

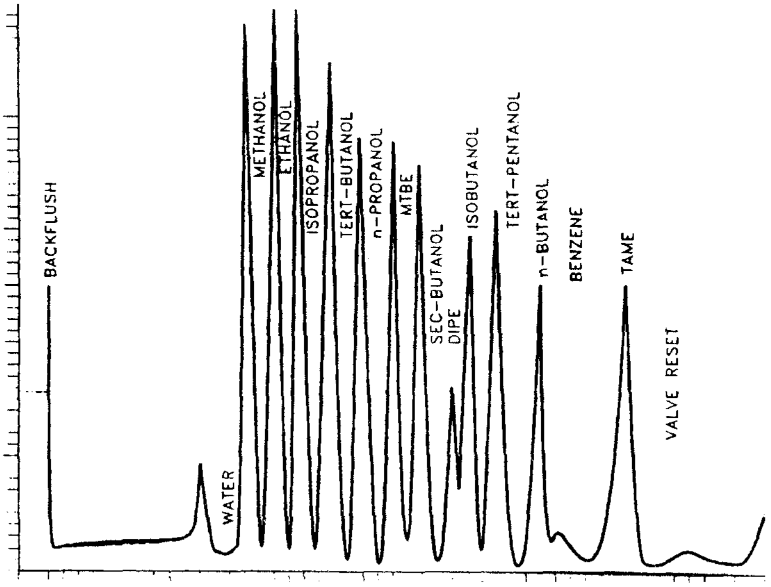


FIG. 3 ANALYSIS OF OXYGENATES IN GASOLINE EXAMPLE CHROMATOGRAM SHOWING OXYGENATES

Completely dissolve 10 g of TCEP in 100 ml of methylene chloride. Next add 40 g of 80/100 mesh Chromosorb P (AW) to the TCEP solution. Quickly transfer this mixture to drying dish, in a fume hood, without scraping any of the residual packing from the sides of the container constantly, but gently, stir the packing until all of the solvent has evaporated. This column packing can be used immediately to prepare the TCEP column.

C-8 PREPARATION OF MICRO-PACKED TCEP COLUMN

C-8.1 Wash a straight 560 mm length of 1.6 mm outside diameter (0.38 mm inside diameter) stainless steel tubing with methanol and dry with compressed nitrogen.

C-8.2 Insert 6 to 12 strands of silvered wire, a small mesh screen or stainless steel frit inside one end of the tube. Slowly add 0.14 to 0.15 g of packing material to the column and gently vibrate to settle the packing inside the column. When strands of wire are used to retain the packing material inside the column, leave 6.0 mm of space at the top of the column.

C-8.3 Column Conditioning

Both the TCEP and WCOT columns are to be briefly conditioned before use. Connect the columns to the valve (*see* **C-10.1**) in the chromatographic oven. Adjust the carrier gas flows as in **C-10.3** and place the valve in the RESET position. After several minutes, increase the column oven temperature to 120°C and maintain these conditions for 5 to 10 min, cool the columns below 60°C before shutting off the carriers flow.

C-9 SAMPLING

Gasoline samples to be analysed by this test method shall be sampled using procedures stipulated in IS 1447 (Part 1).

C-10 PREPARATION OF APPARATUS AND ESTABLISHMENT OF CONDITIONS

C-10.1 Assembly

Connect the WCOT column to the valve system using low volume connectors and narrow bore tubing. It is important to minimize the volume of the chromatographic system that comes in contact with the sample, otherwise peak broadening will occur.

C-10.2 Adjust the operating conditions to those listed in Table 3, but do not turn on the detector circuits. Check the system for leaks before proceeding further.

C-10.3 Flow Rate Adjustment

C-10.3.1 Attach a flow measuring device to the column vent with the valve in the RESET position and adjust the pressure to the injection port to give 5.0 ml/min flow. Soap bubble flow meters are suitable.

C-10.3.2 Attach a flow measuring device to the split injector vent and adjust the flow from the split vent using the *A* (*see* Fig. 2) flow controller to give a flow of 70 ml/min. Recheck the column vent flow set in **C-10.3.1** and adjust, if necessary.

C-10.3.3 Switch the valve to the BACKFLUSH position and adjust the variable restrictor to give the same column vent flow set in **C-10.3.1**. This is necessary to minimize flow changes when the valve is switched.

C-10.3.4 Switch the valve to the inject position RESET and adjust the *B* (*see* Fig. 2) flow controller to give a flow of 3.0 to 3.2 ml/min at the detector exit. When required for the particular instrumentation used, add makeup flow or TCD switching flow to give a total of 21 ml/min at the detector exit.

C-10.4 When a thermal conductivity detector is used, turn on the filament current and allow the detector to equilibrate. When a flame ionization detector is used, set the hydrogen and air flows and ignite the flame.

C-10.5 Determination of Time to Backflush

The time to backflush will vary slightly for each column system and must be determined experimentally as follows. The start time of the integrator and valve timer must be synchronized with the injection to accurately reproduce the backflush time.

C-10.5.1 Initially assume a valve BACKFLUSH time of 0.23 min. With the valve RESET, inject 3 microlitre of a blend containing at least 0.5 percent of greater oxygenates (*see* **C-6.3**), and simultaneously begin timing the analysis. At 0.23 min, rotate valve to the BACKFLUSH position and leave it there until the complete elution of benzene is realized. Note this time as the RESET time, which is the time at which the valve is returned to the RESET position. When all of the remaining hydrocarbon is backflushed the signal will return to a stable baseline and the system is ready for another analysis. The chromatogram should appear similar to that illustrated in Fig. 3.

C-10.5.2 It is necessary to optimize the valve BACKFLUSH time by analyzing a standard blend containing oxygenates. The correct BACKFLUSH time is determined experimentally by using valve switching times between 0.2 and 0.3 min, when the valve is switched too soon, C_5 and lighter hydrocarbons are backflushed and are co-elluted in the C_4 alcohol section of the chromatogram. When the valve BACKFLUSH is switched too late, part of all of the ether components (MTBE, ETBE or TAME) is vented resulting in an incorrect MTBE measurement. DTPE may require a BACKFLUSH time slightly shorter than the other ethers. The system may require reoptimization, if analysis of DIPE is required.

C-11 CALIBRATION AND STANDARDIZATION

C-11.1 Identification

Determine the retention time of each component by injecting small amounts either separately or in known mixtures or by comparing the relative retention times with those in Table 4.

C-11.1.1 In order to ensure minimum interference from hydrocarbons, it is strongly recommended that a fuel devoid of oxygenates be chromatographed to determine the level of any hydrocarbon interference.

C-11.2 Preparation of Calibration Samples

Prepare multi-component calibration standards of the oxygenates and concentration ranges of interest by mass. For each oxygenate, prepare a minimum of five calibration standards spanning the range of the oxygenate in the samples. As an example, for full range calibration, 0.1, 0.5, 2, 5, 10, 15 and 20 mass percent of each oxygenate may be used. Before preparing the

Table 4 Retention Characteristics for TCEP/ WCOT Column
(Set Conditions as in Table 3)

(*Clause* C-11.1)

Sl No.	Component	Retention Time, min	Relative Retention Time	
			(MTBE = 1.00)	(DME = 1.00)
(1)	(2)	(3)	(4)	(5)
i)	Methanol	3.15	0.63	0.46
ii)	Ethanol	3.48	0.69	0.51
iii)	Isopropanol	3.83	0.76	0.56
iv)	tert-Butanol	4.15	0.82	0.61
v)	*n*-Propanol	4.56	0.90	0.67
vi)	MTBE	5.04	1.00	0.74
vii)	sec-Butanol	5.36	1.06	0.79
viii)	DIPE	5.76	1.14	0.85
ix)	Isobutanol	6.00	1.18	0.88
x)	ETBE	6.20	1.23	0.91
xi)	tert-pentanol	6.43	1.28	0.95
xii)	DME	6.80	1.35	1.00
xiii)	*n*-Butanol	7.04	1.40	1.04
xiv)	Benzene	7.41	1.47	1.09
xv)	TAME	8.17	1.62	1.20

standards, determine the purity of the oxygenate stocks and make corrections for the impurities found. Whenever possible, use stocks of at least 99.9 percent purity. To minimize evaporation of light components, chill all chemicals and gasoline used to prepare standards. Prepare standards by transferring a fixed volume of oxygenates using pipettes or eye droppers (for volumes below one volume percent) to 100 ml volumetric flasks or septum capped vials as follows. Cap and record the tare weight of the volumetric flask or vial to 0.1 mg. Remove the cap and carefully add the oxygenate to the flask or vial. Do not contaminate with sample the part within the flask or vial which contacts the cap. Cap and record the net mass (M_i) to 0.1 mg of the oxygenate added. Repeat the addition and weighing procedure for each oxygenates of interest. Similarly, add 5 ml of the internal standard (DME) and record the net mass (M_s) to 0.1 mg. Dilute each standard to 100.0 ml with oxygenate-free gasoline or a mixture of hydrocarbons such as isooctane/mixed xylene (66.35 volume percent). Do not exceed 30 volume percent for all oxygenates, including the internal standard added. Store the capped calibrations standards below 5°C when not in use.

C-11.3 Standardization

The area under each peak in the chromatogram is considered a quantitative measure of the corresponding compound. Measure the peak area of each oxygenate and of the internal standard by either manual methods or electronic integrator. Calculate the relative volume response factor of each oxygenate, relative to the internal standard.

C-12 PROCEDURE

C-12.1 Preparation of Sample

Transfer 0.5 ml of internal standard (M_s) by a volumetric pipette into a tared and capped 10 ml, volumetric flask. Record weight to nearest 0.1 mg. Record the net mass of the internal standard. Retare the capped flask. Fill the 10 ml volumetric flask to volume with sample, cap and record the net mass (M_g) to the nearest 0.1 mg of the sample added. Mix thoroughly and inject into the gas chromatography. If using an automatic sample then transfer an aliquot of the solution into a glass gas chromatographic (GC) vial. Seal the GC vial with Teflon-lined septum. If the sample is not immediately analyzed, store below 5°C.

C-12.2 Chromatographic Analysis

Introduce a representative aliquot of the sample, containing internal standard into the chromatograph using the same technique as used for the calibration analysis. An injection volume of 3 litre with a 15:1 split ratio has been used successfully.

C-12.3 Interpretation of Chromatogram

Compare the results of sample analysis to those of calibration analysis to determine identification of oxygenates present.

C-13 CALCULATION

C-13.1 Mass Concentration of Oxygenates

After identifying the various oxygenates, measure the area of each oxygenate peak and that of the internal standard. From the least-squares fit calibrations, as depicted in the MTBE example in Fig. 4. Calculate the mass of each oxygenate (M_i) in the gasoline samples using response ratio (rsp_i) of the areas of the oxygenate to that of the internal standard as follows:

$$rsp_i = (m_i)\,(amt_i) + b_i$$

where

m_i = slope of the linear fit,
b_i = y-intercept, and
amt_i = amount ratio that is (M_i/M_s),

or

$amt_i = (M_i)/M_s = (rsp_i - b_i)/m_i$

or

$M_i = [(rsp_i - b_i)/m_i]\,M_s = [(A_i/As - b_i)]M_s$

where

M_i = mass of oxygenate, and
M_s = mass of internal standard.

To obtain mass percent (w_i) results for each oxygenate:

$$w = \frac{M_i\,(100\text{ percent})}{M_g}$$

where

M_g = mass of gasoline sample.

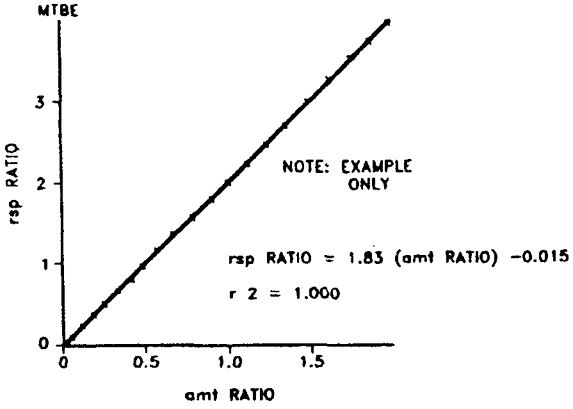


FIG. 4 LEAST-SQUARES FIT CALIBRATION FOR MTBE

C-13.2 Report the mass content of each oxygenate to the nearest 0.01 mass percent.

C-14 PRECISION AND BIAS

C-14.1 Precision

The precision of this test method as determined by the statistical examination of the inter-laboratory test results is as follows.

C-14.1.1 *Repeatability*

The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty (*see* Table 5).

Repeatability Estimates for Oxygenates in Gasoline

Component	*Repeatability*
Methanol	0.09 (*X* 0.59)
Ethanol	0.06 (*X* 0.61)
Isopropanol	0.04 (*X* 0.56)
tert-Butanol	0.04 (*X* 0.56)
n-Propanol	0.003(*X* 0.57)
MTBE	0.05 (*X* 0.56)
sec-Butanol	0.003(*X* 0.61)
DIPE	0.08 (*X* 0.56)
Isobutanol	0.08 (*X* 0.56)
ETBE	0.05 (*X* 0.82)
tert-Pentanol	0.04 (*X* 0.61)
n-Butanol	0.06 (*X* 0.61)
TAME	0.05 (*X* 0.70)
Total oxygen	0.02 (*X* 1.26)

where

X is the mean mass percent of the component.

C-14.1.2 *Reproducibility*

The difference between two single and independent results obtained by different operators working in different laboratories on identical material would in the long run, exceed the following values only in one case in twenty (*see* Table 5).

Table 5 Precision Intervals — Determination from Cooperative Study Data

(*Clauses* C-14.1.1 *and* C-14.1.2)

Component Weight, Percent	**Repeatability**													
	MEOH	EtOH	iPA	tBA	nPA	MTBE	sBA	DIPE	iBA	ETBE	tAA	nBA	TAME	O_2
0.20	0.04	0.02	0.02	0.02	0.01	0.02	0.01	0.03	0.03	0.01	0.02	0.02	0.02	
0.50	0.06	0.04	0.03	0.03	0.02	0.03	0.02	0.05	0.05	0.03	0.03	0.04	0.03	
1.00	0.09	0.06	0.04	0.04	0.03	0.05	0.03	0.08	0.08	0.05	0.04	0.06	0.05	
2.00	0.14	0.09	0.06	0.06	0.05	0.07	0.05	0.12	0.12	0.09	0.06	0.09	0.08	0.02
3.00	0.17	0.12	0.07	0.07	0.06	0.09	0.06	0.15	0.15	0.12	0.08	0.12	0.11	0.05
4.00	0.20	0.14	0.09	0.09	0.07	0.11	0.07	0.17	0.17	0.16	0.09	0.14	0.13	0.08
5.00	0.23	0.16	0.10	0.10	0.08	0.12	0.08	0.20	0.20	0.19	0.11	0.16	0.15	0.12
6.00	0.26	0.18	0.11	0.11	0.08	0.14	0.09	0.22	0.22	0.22	0.12	0.18	0.17	0.15
10.00	0.35	0.24	0.15	0.15	0.11	0.18	0.12	0.29	0.29	0.33	0.16	0.24	0.25	
12.00	0.39	0.27	0.16	0.16	0.12	0.20	0.14	0.32	0.32	0.38	0.18	0.27	0.29	
14.00						0.22		0.35		0.44			0.32	
16.00						0.24		0.38		0.49			0.35	
20.00						0.27		0.43		0.58			0.41	

Component Weight, Percent	**Reproducibility**													
	MEOH	EtOH	iPA	tBA	nPA	MTBE	sBA	DIPE	iBA	ETBE	tAA	nBA	TAME	O_2
0.20	0.14	0.09	0.14	0.07	0.04	0.04	0.15	0.14	0.14	0.11	0.06	0.09	0.14	
0.50	0.24	0.16	0.26	0.12	0.07	0.08	0.28	0.26	0.26	0.21	0.10	0.15	0.22	
1.00	0.37	0.23	0.42	0.19	0.14	0.12	0.44	0.42	0.42	0.46	0.15	0.22	0.31	
2.00	0.57	0.34	0.67	0.30	0.16	0.19	0.70	0.67	0.67	0.61	0.22	0.33	0.44	0.09
3.00	0.72	0.43	0.80	0.40	0.21	0.25	0.92	0.88	0.88	0.83	0.28	0.41	0.54	0.22
4.00	0.86	0.51	1.06	0.48	0.24	0.30	1.11	1.06	1.06	1.03	0.33	0.49	0.63	0.36
5.00	0.99	0.58	1.23	0.56	0.28	0.35	1.29	1.23	1.23	1.22	0.38	0.55	0.70	0.52
6.00	1.10	0.64	1.40	0.63	0.31	0.40	1.46	1.40	1.40	1.41	0.42	0.61	0.77	0.70
10.00	1.51	0.86	1.97	0.89	0.41	0.56	2.06	1.97	1.97	2.07	0.56	0.82	1.00	
12.00	1.68	0.95	2.22	1.00	0.45	0.63	2.33	2.22	2.22	2.38	0.62	0.91	1.10	
14.00						0.70		2.46		2.68			1.19	
16.00						0.77		2.69		2.96			1.28	
20.00						0.89		3.13		3.51			1.43	

Reproducibility Estimates for Oxygenates in Gasoline

Component	*Reproducibility*
Methanol	0.37 (X 0.59)
Ethanol	0.23 (X 0.57)
Isopropanol	0.42 (X 0.67)
tert-Butanol	0.19 (X 0.67)
n-Propanol	0.11 (X 0.57)
MTBE	0.44 (X 0.67)
sec-Butanol	0.42 (X 0.67)
DIPE	0.42 (X 0.67)
Isobutanol	0.42 (X 0.67)
ETBE	0.36 (X 0.76)
tert-Pentanol	0.15 (X 0.57)
n-Butanol	0.22 (X 0.57)
TAME	0.31 (X 0.51)
Total oxygen	0.09 (X 1.27)

where

X is the mean mass percent of the component.

C-14.2 Bias

The National Institute of Standards and Technology (NIST) provide selected alcohol in reference fuels. As an example reference materials (SRM) in reference fuels are available as described in the NIST Standard Reference Catalogue (*see* Table 6).

Table 6 Reference Materials

(*Clause* 14.2)

Sl No. (1)	SRM (2)	Type (3)	Nominal MeOH (4)	Concentration, EtOH (5)	Mass Percent of MeOH + t-BuOH (6)
i)	1829	Alcohols in reference fuel	0.355	11.39	10.33 + 6.63
ii)	1837	Methanol and tert-butanol			10.33 + 6.33
iii)	1828	Ethanol		11.39	
iv)	1829	Methanol	0.335		

ANNEX D

[(*Table* 1, *Sl No.* (xxi)]

ADDITIONAL CALCULATIONS

D-1 OXYGENATES CONTENT BY VOLUME

For each component, calculate the oxygenate content in the sample, C_1 as a percentage by volume according to the following equation:

$$C_1 = \frac{C_0 P_1}{P_2}$$

where

C_0 = content of the component calculated according to **C-13.1** (percent);

P_1 = density of the gasoline sample, g/ml; and

P_2 = density of the component, g/ml, as given in Table 7.

D-2 TOTAL OXYGEN CONTENT

D-2.1 For each component calculate the oxygen content in the sample C_2, as a percentage by mass according to the following equation:

$$C_2 = \frac{C_0 \times 16}{M}$$

where

C_0 = content of the component calculated according to **C-13.1** (percent); and

M = relative molecular mass of the component as given in Table 7.

D-2.2 Calculate the total oxygen content of oxygenates in the gasoline sample by summing the individual oxygen content C_2 (*see* **D-2.1**) for all components present.

Table 7 Physical Data for Oxygenates

(*Clauses* D-1 *and* D-2.1)

Sl No. (1)	Component (2)	Relative Molecular Mass (3)	Density, g/ml at 20°C (4)
A	**Alcohols**		
i)	Methanol	32	0.791
ii)	Ethanol	46	0.793
iii)	Propan-2-ol	60	0.786
iv)	Butan-l-ol	74	0.810
v)	t-butyl alcohol	74	0.789
vi)	isobutyl alcohol	74	0.802
B	**Ethers**		
i)	Methyl tertiary butyl ether	88	0.740
ii)	Ethyl tertiary butyl ether	102	0.752
iii)	Methyl tertiary amyl ether	102	0.770

ANNEX E

[*Table* 1, *Sl No.* (xxii) (b), *Note* 8]

METHOD FOR DETERMINATION OF ETHANOL CONTENT IN 5 PERCENT ETHANOL DOPED MOTOR GASOLINE BY WATER EXTRACTION

E-1 GENERAL

This method covers the determination of ethanol content in ethanol doped motor gasoline as field test at supply point, retail outlet etc. by using water. This method provides a test of on line ethanol doped gasoline for ethanol content by percent by volume. The method can suitably be used for estimation of ethanol content in ethanol doped motor gasoline and quantification 5 percent ethanol (up to 8 percent) can be made by using the method in down stream. In case of dispute the sample may be subjected to ASTM D 4815 method.

E-2 OUTLINE OF THE TEST METHOD

30 ml of water is added to 100 ml of ethanol doped motor gasoline taken in 100 ml graduated stoppered cylinder. The mixture is shaken vigorously for about 30 s and cylinder is set aside for 5 min to observe the separation of aqueous layer. Increase in the volume of aqueous layer over and above 30 ml indicates the presence of extractable ethanol in the ethanol doped motor gasoline sample tested. The exact quantity of ethanol is then worked out referring the observation to the calibration plot. The plot is made on exact quantity of ethanol extracted from 100 ml of various known concentrations of ethanol doped motor gasoline using 30 ml of water vis-à-vis the concentration of ethanol used for doping. For better results the calibration chart shall be re-plotted whenever there is change in the source of motor gasoline.

E-3 SIGNIFICANCE AND USE

The test method is employed to check the percentage of ethanol in ethanol doped motor gasoline, as ethanol is having more affinity for mixing with water. The addition of water in ethanol doped motor gasoline extracts the ethanol present in the ethanol doped motor gasoline in to aqueous phase. The increase in the water level indicates the quantity of extractable ethanol in ethanol doped motor gasoline.

E-4 CHEMICAL REQUIREMENTS

E-4.1 Ethanol/Denatured Ethanol, is the pure chemical, otherwise known as hydroxy-ethane, corresponding to the constitution CH_3CH_2OH and molecular formula, C_2H_5OH. Nominally anhydrous ethanol (*see* IS 15424) made unfit for beverage use by the addition of noxious or toxic materials (denaturants) and same is used as ethanol for the purpose of doping motor gasoline.

E-4.2 Motor Gasoline, a volatile mixture of liquid hydrocarbon, generally containing a small amount of additives, suitable for use as a fuel in spark ignition and internal combustion engines conforming to Table 1.

E-4.3 Ethanol Doped Motor Gasoline, a fuel consisting primarily of motor gasoline doped with 5 percent (*v/v*) of denatured anhydrous ethanol for fuel.

E-4.4 Water, distilled is preferred for extraction procedure, but potable water may also be used.

E-5 APPARATUS

E-5.1 100 ml Glass Stoppered Cylinder with 1 ml Graduation — The distance between 100 ml mark and the top of the shoulder of the cylinder shall be above 40 ml.

E-5.2 Graduated Pipette — 1 ml to 10 ml.

E-6 CALIBRATION ON KNOWN ETHANOL CONTENT IN ETHANOL DOPED MOTOR GASOLINE AND ITS EXTRACTION

E-6.1 Prepare a set of standards of ethanol doped motor gasoline samples with the concentration of ethanol in motor gasoline ranging from 0.5 to 8 ml by percentage volume. Take 100 ml of each sample above in 100 ml graduated stoppered clean and dry glass cylinder along with one blank motor gasoline (without ethanol). Add precisely 30 ml water to the sample. Shake the stopper cylinder vigorously for at least for 30 s. Loosen the stopper of glass cylinder to release the vapour pressure in the cylinder but do not remove the stopper. Tighten the stopper and allow the stopper cylinder to sit undisturbed at ambient temperature upright on the leveled surface for a period of at least 5 min to allow the water-ethanol mixture to settle to the bottom.

E-6.2 If separation of two layers is not complete, lightly tap the cylinder to encourage the complete separation. Record the level of aqueous layer in glass cylinder by reading the measurement at the lowest part of the meniscus.

> Presence of ethanol percent in ethanol doped motor gasoline = Volume of aqueous layer observed in graduated cylinder – 30 ml (water added during testing).

E-6.3 Plot a graph on various values obtained above as ordinate (X-axis) against corresponding concentration of ethanol used in the standard blend as abscissa (Y-axis) including that of the observation made on the blank.

E-7 PROCEDURE

E-7.1 Take 100 ml sample of ethanol-blended motor gasoline under test in 100 ml graduated stoppered clean and dry glass cylinder. Add precisely 30 ml water to the sample taken in the glass cylinder and place the stopper tightly. Shake the stoppered cylinder vigorously for at least 30 s. Loosen the stopper of glass cylinder to release the vapour pressure in the cylinder but do not remove the stopper. Tighten the stopper and allow the stoppered cylinder to sit undisturbed at ambient temperature upright on the leveled surface for a period of at least 5 min to allow the water-ethanol mixture to settle to the bottom.

E-7.2 If separation of two layers is not complete, lightly tap the cylinder to encourage the complete separation. Record the level of aqueous layer in glass cylinder by reading the measurement at the lowest part of the meniscus.

> Presence of ethanol percent, in ethanol doped motor gasoline = Volume of aqueous layer observed in graduated cylinder – 30 ml (water added during testing).

E-8 CALCULATION

Observation made under **E-7.2**, is read against the standard plot in **E-6.3** and the corresponding concentration of ethanol value is obtained. Whenever there is a change in source of motor gasoline, for better results, it is recommended to re-plot the graph as mentioned in **E-6.3**.

E-9 REPORT

Report the corrected results obtained as per **E-8** to the nearest 0.5 ml.

E-10 PRECISION AND BIAS

E-10.1 In the absence of calibration plot the value observed in aqueous phase can be calculated to the nearest percentage volume as per the under mentioned precision levels.

E-10.2 Based on the field test data, it was observed that a maximum of 4.6 ml (average) of ethanol content is extracted in to the aqueous phase as against 5 ml ethanol added in ethanol doped motor gasoline.

E-10.3 The standard deviation of the test results indicates ± 0.24 percent.

E-10.4 Considering the precision of the graduated glass cylinder, the maximum possible measurement can be treated as 4.5 percent volume with standard deviation of ± 0.5 percent. Thus the repeatability under the method is as under:

Percentage	*Repeatability / Reproducibility*
5 percent (*v/v*)	0.5ml (Approximately)

ANNEX F

(Foreword)

EURO IV/BHARAT STAGE III EMISSION NORMS COMPLIANT SPECIFICATION FOR MOTOR GASOLINE (REQUIREMENTS)

Sl No.	Characteristics	Requirements		Method of Test, Ref to [P :] of IS 1448/Annex of IS 2796/ISO/Alternate Methods
(1)	(2)	(3)	(4)	(5)
i)	Grade	MG 91	MG 95	
ii)	Colour, visual	As decided by refiners/marketers		—
iii)	Density @ 15°C, kg/m^3	720-775	720-775	[P : 16]1)/ ISO 3675/ (ASTM D 4052)
iv)	Distillation:			[P : 18]1)/ ISO 3405/(ASTM D 86)
	a) Recovery up to 70°C (E-70), percent by volume	10-45	10-45	
	b) Recovery up to 100°C (E-100), percent by volume	40-70	40-70	
	c) Recovery up to 150°C (E-150), percent by volume, *Min*	75	75	
	d) Recovery up to 180°C (E-180), percent by volume, *Min*	—	—	
	e) Final boiling point, °C *Max*	210	210	
	f) Residue, percent by volume, *Max*	2	2	
v)	Research octane number (RON), *Min*	91	95	[P : 27]/ISO 5164/ASTM D2699
vi)	Motor octane number (MON), *Min*	81	85	[P : 26]/ISO 5163/ASTM D2700
vii)	Existent gum, g/m^3, *Max*	40	40	[P : 29]/ISO 6246/ASTM D 381
viii)	Sulphur, total, mg/kg *Max*	50	50	[P : 34]1)/ASTM D 1266/[P:83], ISO 4260, D2785, D 5453, D 2622, ISO 14596, D 3120, ISO 20847
ix)	Lead content (as Pb), g/l, *Max*	0.005	0.005	[P : 80]1)/IP 224/IP 352/ (ASTM 5059)
x)	Reid vapour pressure (RVP) at 38°C, kPa (*see* Note 2)	60 (67) *Max*	60 (67) *Max*	[P : 39]1)/ISO 3007/ASTM D 323/ (EN 13016, ASTM D 5191, Annex A) (*see* Note 3)
xi)	Vapour lock index (VLI), (VLI=10 RVP + 7E 70), *Max* (*see* Notes 2 and 3)			
	a) Summer (*see* Note 5)	750 (900)	750 (900)	
	b) Other months	950 (1 050)	950 (1 050)	
xii)	Benzene content, percent by volume, *Max*	1	1	ASTM D 3606 (ASTM D 5580, D 6277, D 6730 (*see* Note 4)
xiii)	Copper strip corrosion (for 3 h @ 50°C), *Max*	Not more than 1	Not more than 1	[P : 15]/ISO 2160/ASTM D 130
xiv)	Water tolerance of gasoline-alcohol blends, temperature for phase separation, °C, *Max*			Annex B
	a) Summer	10	10	
	b) Winter (*see* Note 6)	0	0	
xv)	Engine intake system cleanliness (*see* Note 7)	Report-MFA Used	Report-MFA Used	—
xvi)	Olefin content, percent by volume, *Max*	21	18	[P : 23]1)/D 1319/ISO 3738/ (ASTM D 6730)
xvii)	Oxidation stability, *Min*	360	360	[P : 28]/(ISO 7536/ASTM D 525/IP 40)
xviii)	Aromatic content, percent by volume, *Max*	35	35	[P : 23]/ASTM D 1319/ASTM D 6730/ISO 3738/(ASTM D 5580, D 6730)
xix)	Oxygen content, percent by mass, *Max*	2.7	2.7	EN 1601/IP 408/ASTM D 4815
xx)	Oxygenates, percent by volume, *Max*			ASTM D 4815/Annex C
	a) Methanol	Nil	Nil	

ANNEX F (*Concluded*)

b)	Ethanol (*see* Note 8)	5	5
c)	Iso-Propyl alcohol	10	10
d)	Iso-butyl alcohol	10	10
e)	Tertiary-butyl alcohol	7	7
f)	Ethers containing 5 or more 'C' atoms per molecules	15	15
g)	Other oxygenates (*see* Note 9)	8	8

NOTES

1 Limits given in ( ) shall be applicable to 5 percent (*v/v*) ethanol-blended gasoline only. Those areas where blending of ethanol in gasoline is compulsory the dosage of ethanol shall be 4.75 ± 0.25 (*v/v*).

2 Summer shall be the period from May to July.

3 In winter it is expected that temperature may be lower than 0°C in the northern hilly region and hence phase separation shall not take place till –10°C.

4 Use of multifunctional additives (MFA) is a requirement for assuring adequate fuel system and intake system cleanliness performance in engines. Refiners/Marketers of motor gasoline have to ensure the MFA[2] has proper credentials from internationally accepted test laboratories/authorities, of having passed a minimum of one of the tests in each of the two categories of deposit control performance indicated below :

	Deposit Type	*Test No.*	*Test Equipment*	*Test Method*	*Pass Limit (Average IVD) (Weight)*
a)	Intake valve deposit (IVD)	1	BMW IVD Test	ASTM D 5500-97	100 mg/valve, *Max*
		2	MB M102E IVD	CEC F-05-A-93	50 mg/valve, *Max*
		3	FORD 2.3 Litre IVD	ASTM D 6201	90 mg/valve, *Max*
		4	MB M111-IVD Test	CEC F-20-A-98	50 mg/valve, *Max*
					Limit (percent injector flow loss) Pass limit (percent injector flow loss)
b)	Port fuel injector Deposit (PFI)	1	Chysier PFI test	ASTM D 5598-95 A	5, *Max*
		2	PFI deposit rig	ASTM D 6421	10 percent, *Max*

Other performance tests may be added as and when they reach qualified/standard test status.

5 Stabilizing agents may be added. For determination of ethanol content ASTM D 5599 or ASTM D 4815 shall be employed. For routine analysis, Method ASTM D 5845 (FTIR) or/and Water extraction method may be employed (*see* Annex E).

6 Acetone is not permitted except when present as a by-product of the manufacture of certain oxygenate compounds and then only up to 0.8 percent (*v/v*).

7 All the test methods referred to in this standard include a precision statement. The interpretation of results based on test method/precision shall be used whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.

8 Type test for phosphorus content in petrol shall be introduced shortly.

9 For test methods, *see* Table 1 and foreword.

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[1] In case of dispute, this method shall be the referee method.

[2] MFAs which are certified against National Generic Certification Option as per US EPA-97 final rule (40 CFR Part 80 certification standards for deposit control additives) can also be used by fuel refiners/marketers at treatment levels not less than the Lower Additives Concentration (LAC) limits, as these MFAs meet the criteria for acceptance mentioned above.

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